Corrosion

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the revolutionary fiber glass reinforced epoxy pipe for corrosive service

114

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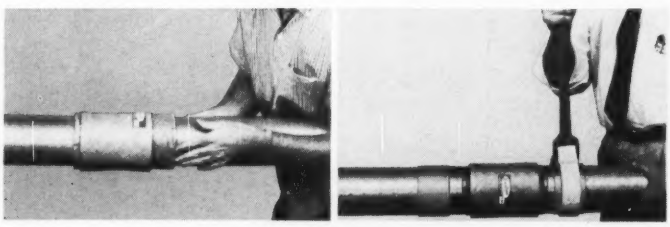
Here's a pipe that holds up indefinitely under the corrosive action of many salt, acid and alkaline solutions . . . that can't contaminate or flavor the piped material . . . that is rigid enough to resist sag, cold flow or deformation – flexible enough to follow normal ditch contours . . . that remains smooth and unclogged throughout its entire service life . . . that won't leak, even near its burst pressure.



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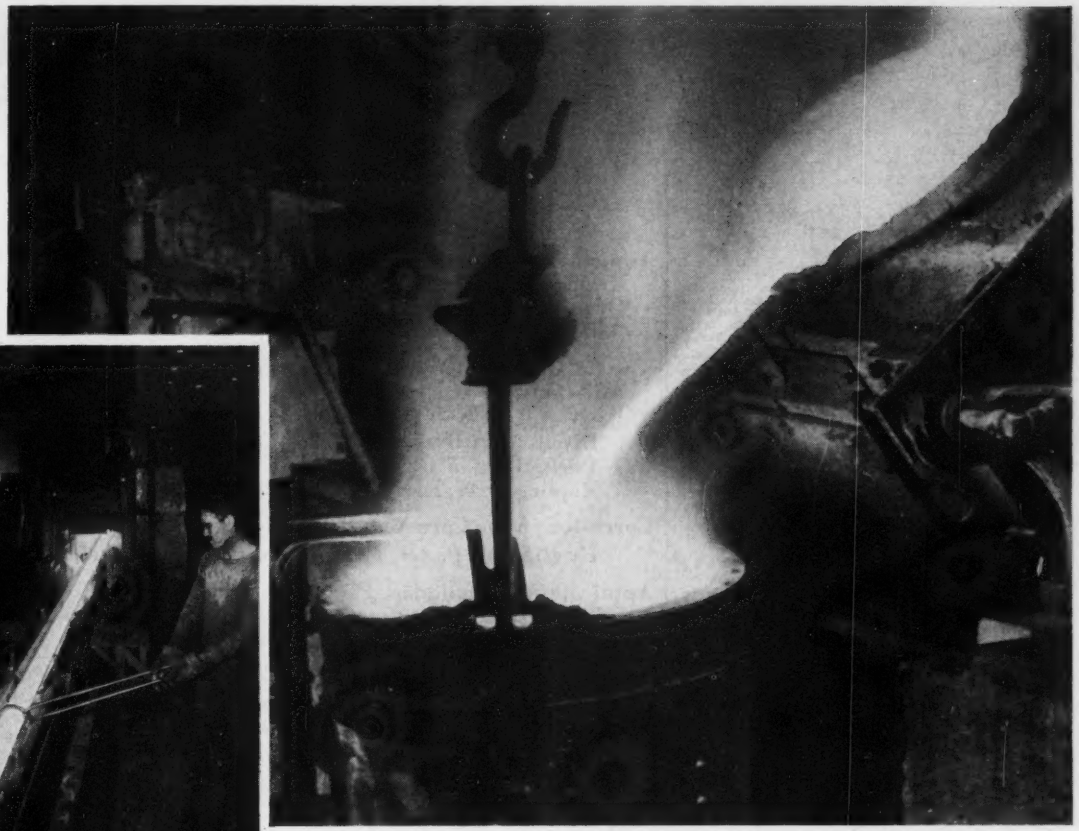

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Official U. S. Navy Photograph

THIS MONTH'S COVER—This cavitation-corrosion area on the aft (pressure) face (suction side) of starboard outboard manganese-bronze propeller on USS Lake Champlain is characteristic of this phenomenon which occurs near the hub in many cases. Pit depths up to 1½-in. in 18 months have been reported. Studies of ways to prevent this damage are actively pursued by the Navy.

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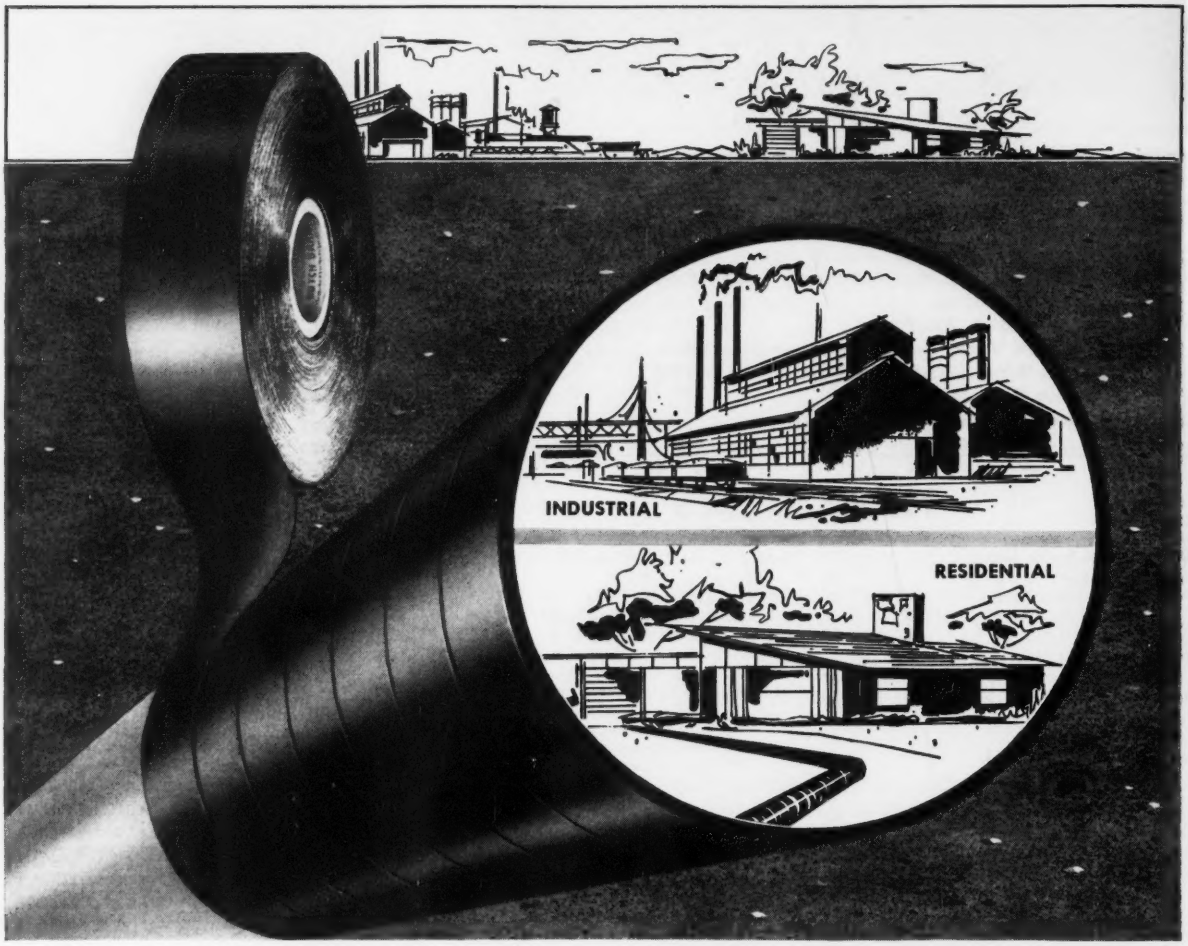

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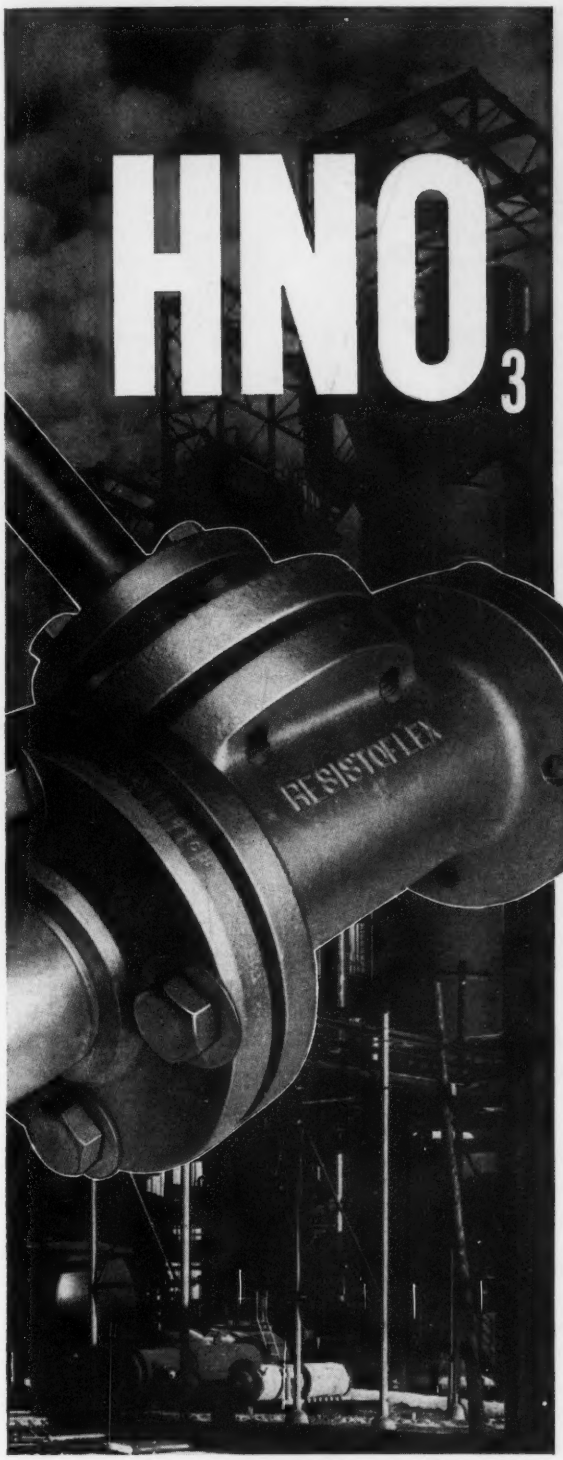

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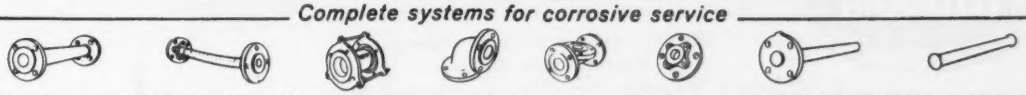

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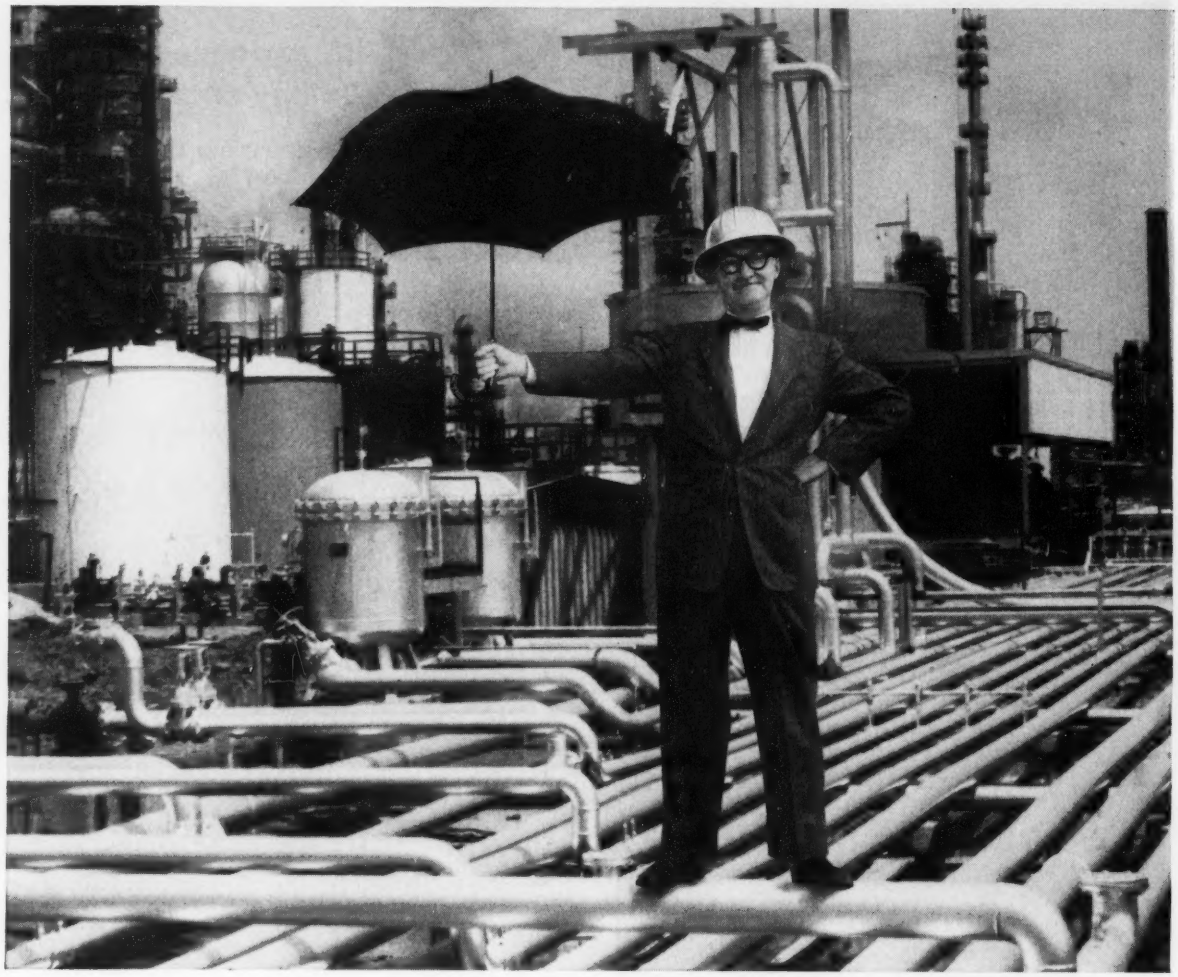

THERE'S A BETTER WAY... to protect refineries, chemical and industrial plants against corrosion!

Corrosion works overtime on any metal surface that isn't properly protected. Positive corrosion control begins with Glidden Protective Maintenance Coating Systems. They provide *real umbrella protection!*

Glidden Protective Maintenance Coatings are designed to solve specific corrosion and erosion problems. They protect steel, galvanized iron, aluminum, as well as various masonry and wood surfaces.

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As part of the Glidden Protective Maintenance Program, our field engineers will analyze your particular problems and recommend the correct system for positive corrosion control at lowest maintenance cost per year.

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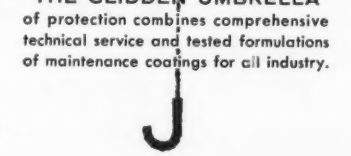

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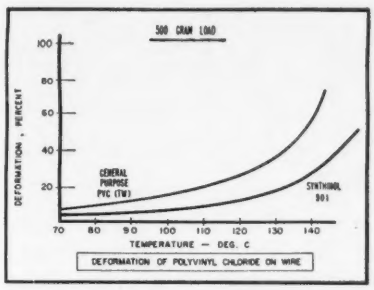
Troubled with wiring failures in your refinery?

If so, consider the record being established by Rome Synthinol 901, a special 90° PVC compound for wire insulation and sheathing. It has helped to solve an impressive list of difficult wiring problems.

Developed by Rome Cable's laboratory and first introduced in 1949, Rome Synthinol 901 has proved by test and actual use to be superior to conventional PVC compounds in the following ways:

a. Suitable for continuous operation at higher temperature (90°C).
b. More stable under severe heat cycling.
c. Suitable for continuous immersion in hot transformer oil.
d. Superior resistance to chemicals and solvents.
e. Greater resistance to heat deformation.



Graph shows heat deformation vs. temperature, and compares Synthinol 901 with conventional PVC compounds. Test run in accordance with Underwriters' Laboratories standard procedure, except for temperature.

The practical value of this compound can best be illustrated by describing a number of actual case histories.

(1) Oil refinery, industrial and chemical plant wiring.

Plant and oil refinery engineers have long used conventional PVC insulations for plant wiring with good results, but too often heat deformation problems arose in warm locations. Since Rome Synthinol 901's superior deformation resistance suggested that it might be the answer to this problem, it was tried and soon became widely used because of its much better service record.

Synthinol 901 also proved to be far superior to conventional materials for chemical plant wiring where the corrosive action of chemicals and solvents has always been a tough problem. Although polyvinyl chlorides as a class have inherently better resistance to chemicals and solvents than many other materials, some engineers desired even further improvement. One user decided to test a number of samples over a long period by immersing them in various concentrations of acetone, different alkalis, and butyric, propionic, and acetic acids. The results of this careful evaluation showed clearly that, when exposed to these chemicals, Synthinol 901 would last longer than any other PVC tested.

(2) Oil-filled transformers.

Transformer engineers have long sought a cable construction to replace varnished cambric-insulated cable for leads in oil-filled transformers. These leads connect transformer windings and bushings and are constantly immersed in hot oil. One transformer manufacturer, encouraged by successful experience with Synthinol 901 on other applications, undertook a comprehensive study of the possible usefulness of this insulation in lieu of varnished cambric for high-voltage transformer leads. This study extended over a several-year period and proved to those engineers involved that there were at least four reasons for standardizing on Synthinol 901 for this application:

a. There was less sludge in the oil than when VC insulation was used.
b. There was no harmful degradation of the electrical properties of the oil.
c. Conductors insulated with Synthinol 901 ran cooler, permitting a 7% increase in lead cable current rating.
d. The use of Synthinol 901 in lieu of VC would permit substantial dollar savings.

(3) Coil leads.

Many manufacturers of small coils for relays, small transformers, small motors, and electronic components have tried to use PVC insulated lead wires for many years. Typically, these leads are crimped or soldered to the ends of the coil; and the whole coil is then impregnated with varnish or wax and baked as long as 16 hours at temperatures as high as 150° C. The varnish used may be asphaltic, phenolic, alkyd, etc.; and the wax may be paraffin, beeswax, carnauba, or another kind.

PVC insulated lead wires frequently produced serious production problems because of the insulation's tendency to shrink, crack, and stiffen during the impregnation and subsequent baking period. Synthinol 901 was substituted in place of the ordinary PVC insulation and, after nearly eight years of wide usage, not a single case of shrinkage, cracking, or stiffening of insulation involving Synthinol 901 has been brought to our attention.

These case histories describe just a few of the applications for which Rome's Synthinol 901 has proved especially effective. Perhaps they will suggest other applications in your own area which might also be better handled with Rome Synthinol 901 instead of a conventional PVC compound. If you have a wire or cable problem that might be solved with Rome Synthinol 901, why don't you get in touch with us? Contact your nearest Rome Cable representative for more information—or write to Department 320, Rome Cable Corporation, Rome, N. Y.

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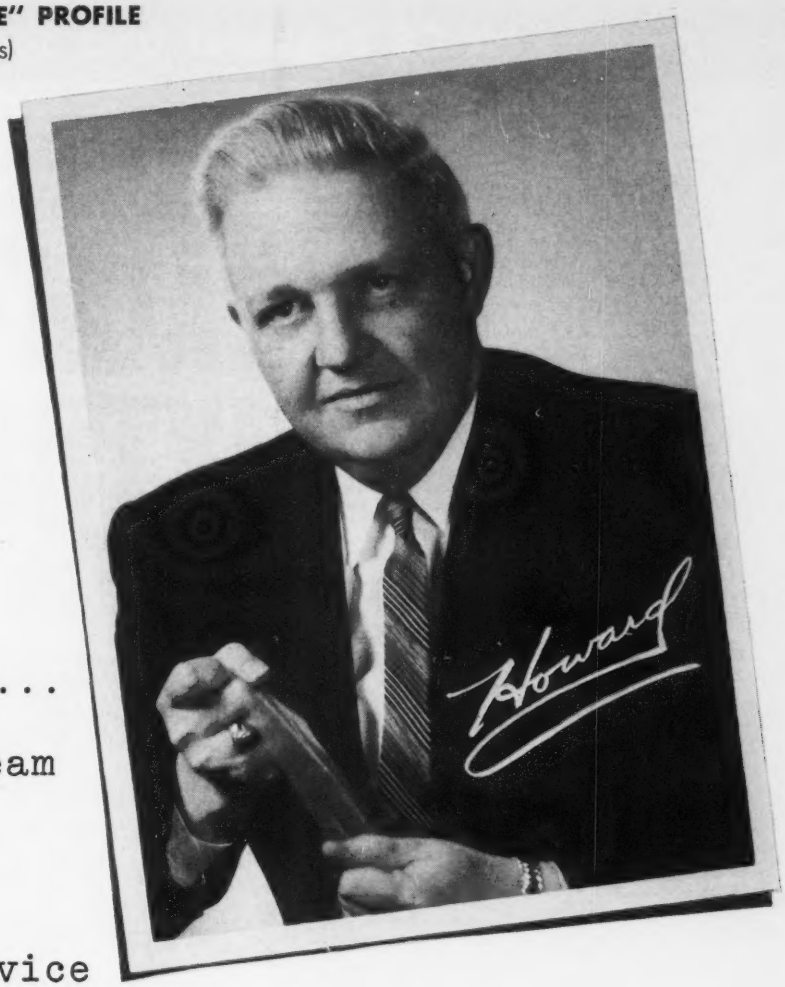

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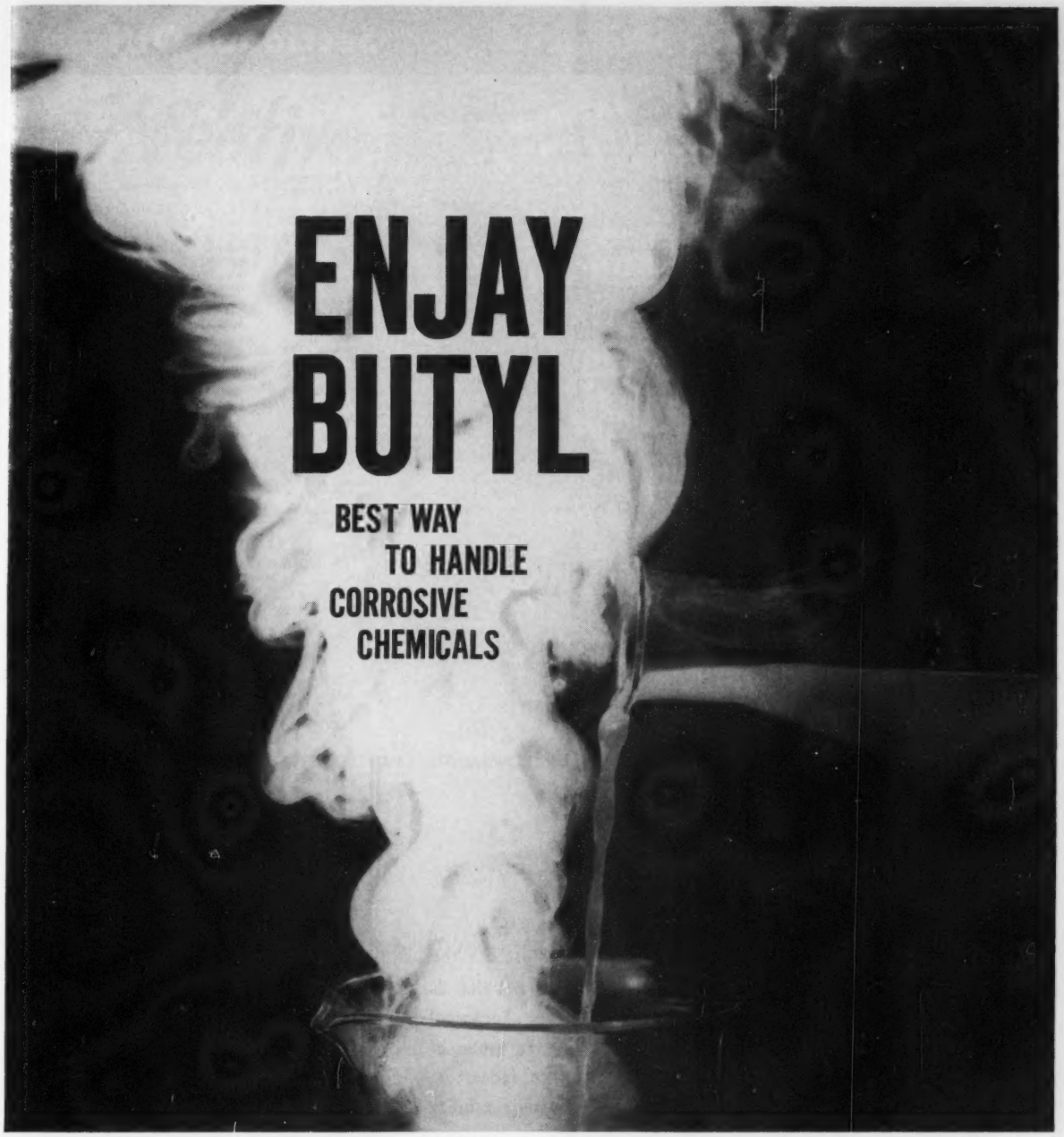

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- By Volumes annually in December issue
- In NACE Bibliographic Surveys of Corrosion
- In NACE Abstract Punch Card Service
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Technical Articles Scheduled for April

Evaluation of Steam Resistant Coatings for Carbon Steel Hospital Ware, by E. G. Babalek, W. R. R. Park, E. G. Bell and W. R. Dawson

Effect of Sigma Phase vs Chromium Carbides on the Intergranular Corrosion of Types 316 and 316L Stainless Steel (Part 1), by Donald Warren

Oil Refinery Applications of Thick-Film Synthetic Coatings, by R. W. Maier, W. B. Cook and R. B. MacQueen

The Delayed Failure of High Strength Steels, by A. R. Troiano

The Action of the XO_4^{n-} Inhibitors, by G. H. Cartledge

Corrosion Problems Associated With Uranium Refining, by F. H. Meyer, Jr., F. J. Podlipec and T. R. Kato

Corrosion Rate Measurement by Hydrogen Effusion in Dynamic Aqueous Systems at Elevated Temperature and Pressure, by M. Krulfeld, M. C. Bloom and R. E. Seebold

Visual Observations on the Water Stream in an Elevated Temperature High Velocity Loop, by R. E. Seebold

Some Experiments on Internal Oxidation of Nickel Chromium Alloys, by H. R. Copson and F. S. Lang

Identification of Corrosion Products on Copper and Copper Alloys—A Report of NACE Technical Unit Committee T-3B on Corrosion Products.

Minimum Corrosion for Butane Isomerization Units—A Contribution to the Work of NACE Technical Group Committee T-8.

Corrosion of Refinery Equipment by Aqueous Hydrogen Sulfide. A Contribution to the Work of Group Committee T-8.

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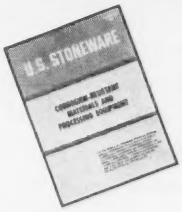

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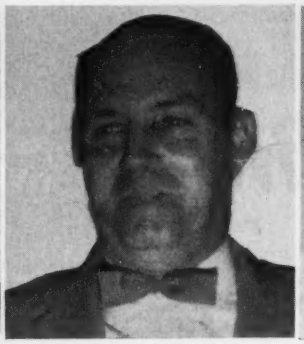

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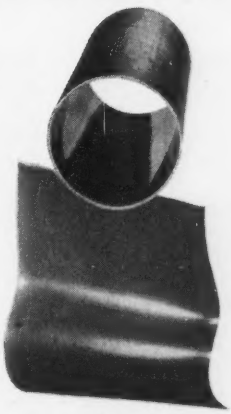

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Corrosion

OFFICIAL MONTHLY PUBLICATION

National Association of Corrosion Engineers

An Association of Individuals and Organizations Concerned with Control of Corrosion

1061 M & M Building Houston 2, Texas

Vol. 15 March, 1959 No. 3

Topic of the Month

Equipment and Procedure for the Evaluation Of Stress Corrosion Susceptibility Of a Wrought Copper Base Alloy

By G. B. SMITH*

Abstract

An account is given of laboratory tests conducted to determine the susceptibility of wrought aluminum bronze alloys to stress corrosion cracking. Equipment used in the tests is described in detail as is testing procedure. Interpretation of results is considered briefly. 2.3.7

Introduction

A SERIES of laboratory tests were conducted to determine the susceptibility of wrought aluminum bronze alloys to stress corrosion cracking. These stress corrosion tests were designed to compare different alloy compositions under the same exposure conditions in the development of a new alloy.

The conditions of the test required equipment which would expose the material under a retained stress in an environment which would promote stress corrosion cracking failure. A description of the equipment, test procedure and interpretation of results are described in this article.

Equipment

Pressure vessels fabricated from 4 inch schedule 80 stainless steel pipe threaded (4-½ inch-12UN-2A) at both ends were used in the experimental work. End covers, threaded (4-½ inch-12UN-2B), were made of stainless steel forged plates and Ampcoloy 45 forged bar stock. This latter alloy was used for the removable end cover to prevent galling.

The stainless steel end cover threaded in place and fillet welded along the top edge was used as the permanent bottom cover plate. Two parallel flats were milled in the outer round of the removable end cover to aid in the securing and removal of the end cover. The inside chamber of the pressure vessels was approximately 3¾ inches in diameter and 9 inches long.

Tubular liners of chemical glass and Teflon were used for certain exposure media. Fluorinated plastic gaskets in the cover sealed the vessel openings to prevent leakage and contact of the corrosive with the end covers. For steam tests and tests in very mild acids or caustics, no liner would be necessary. However, the fluorinated plastic gaskets were used in all tests.

An appropriate size chemical glass beaker was used to hold the liquid for steam tests and most corrosive media except strong caustics and hydrofluoric acid. The latter two agents required stainless steel containers.

Figures 1 and 2 show the pressure vessel and a stress fixture with a specimen in the stressed condition. The stress fixtures were made from ¾ inch 18-8 stainless steel plates and contain a stressing screw of 18-8 stainless steel. Dimensions of the fixtures are shown in Figure 3.

Test Specimens

Test specimens of the wrought copper base material were ⅛-inch thick ½-inch

* Ampco Metal, Inc., Milwaukee, Wisconsin.

Figure 1—Pressure vessel assembly.

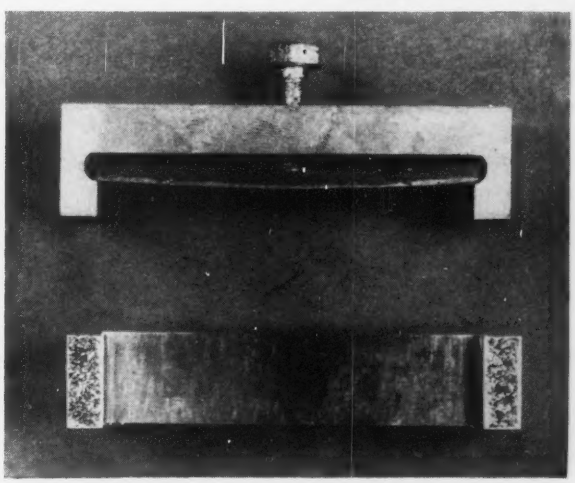

Figure 2—Stress fixture and specimen.

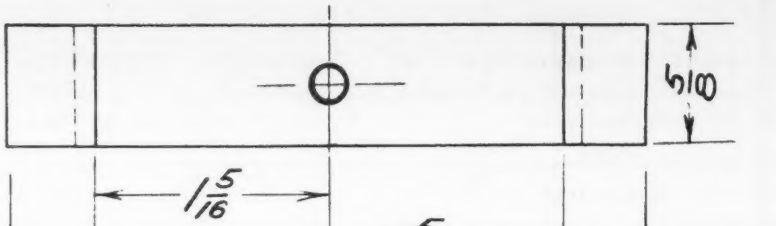

Figure 3—Dimensions of stress fixture.

to ⅝-inch wide and 2⅝-inches long. Specimens which were cut from rolled plate were cut so that the longitudinal axis of the specimen was perpendicular to the rolling fibers, or direction of the final rolling. This specimen orientation will result in the greatest probability of specimen failure. Specimens made from plate or sheet over ⅛-inch thick were milled to ⅛-inch (—.00 + .015) by milling one side. The side which was not machined, (i.e., the rolled surface), was exposed in tension.

All test specimens were thoroughly cleaned, degreased and rinsed with water before being stressed. The edges of the specimens were filed to a small radius, since rough specimen edges tend to promote early failures and yield unsatisfactory test results.

Test Procedure

The test vessel was set upright on the permanent end cover plate and the proper liners inserted where required. The tubular liners, compressed by tightening the cover, contacted the end gaskets securely to form a seal against leakage. A glass or stainless steel container with 250 cc of the liquid was inserted with the bottom of the container resting on the bottom gasket. A corrosion resistant support plate of such size and shape to rest securely was placed across the top of the container. This plate was perforated to allow free passage of vapor and liquid between the top and bottom of the vessel.

The wrought bronze specimens were subjected to a beam type bending stress by the center screw in the stress fixture. Each specimen was stressed at or just above the yield point by turning the center screw 1⅜ turns, producing a center deflection of 0.067 inch in the specimen. The test was based on standard deflection rather than a standard stress level because of the difficulty of accurately calculating the actual stress in the specimen.

Specimens, stressed in the fixtures, were set on the support plate above the liquid for tests performed in the vapor zone. A Teflon gasket was set over the liner or vessel opening and the end cover was screwed securely in place by means of a wrench designed to fit the parallel flats of the end cover.

The vessel was placed in an oven and held at a temperature of 350 F for the duration of a test period. At the conclusion of a test run, the vessel was removed from the oven and allowed to cool in air at least four hours before removal of the end cover.

A five hour period for the vessel and contents to reach temperature and the four hour cooling period was not included as test time.

When the vessel had cooled sufficiently, the end cover was removed and the specimens carefully examined for cracks while in the fixture. Specimens which appeared to be sound after a test run were recharged for additional test runs. A series of test runs of 150 hours duration was used to evaluate each alloy. This time period was selected as an optimum to minimize the errors of exposure time caused by heating and cooling the vessel.

Interpretation of Results

Specimens which survived 1200 hours of testing time, showed no incipient intergranular corrosion attack and were able to withstand a 45 degree bend test without failure were considered to be resistant to stress corrosion cracking in a particular test media.

Small cracks in the bend surface of the specimens were studied and evaluated as mechanical cracks, or incipient stress corrosion cracks.

The coloration of the crack is frequently a good criterion for the determination of the type of crack. Cracks which appear to be the color of the normal metal fracture can be considered mechanical cracks and cracks which show discolored metal may be stress corrosion cracks. However, in either case the material should undergo metallographic examination with a proper etchant to verify the nature of the crack, and to reveal incipient cracking.

Specimens of known susceptibility to stress corrosion cracking were included in each test run. These pilot samples, under stress conditions, failed within the 150 hour test period and served to verify that the test conditions were proper for the evaluation of stress corrosion cracking susceptibility in the various alloys.

Any discussions of this article not published above will appear in the June, 1959 issue

The Corrosion of Zirconium In Hydrochloric Acid at Atmospheric Pressure*

By W. E. KUHN

Abstract

Short term (144 hour) corrosion tests of zirconium subjected to differing surface treatments prior to immersion in aqueous hydrochloric acid solutions suggested that factors involved in film formation play a dominant role in the corrosion behavior of zirconium. Long duration tests (2000 hours) revealed two general types of protective films: one, a protective primordial zirconium oxide film, and the other, a face-centered cubic zirconium monohydride film.

The oxide film is subject to failure by a "breakaway" mechanism involving the formation of an interfacial hydride layer at the oxide-metal interface which eventually causes the protective film to suddenly loosen and flake away. The time for this to occur may vary from less than 144 hours to greater than 2000 hours depending on the integrity of the oxide film. The integrity of the film is in turn dependent on the history of its formation. After "breakaway" the corrosion rate loss in boiling 20 percent hydrochloric acid may range from 3 to a maximum of 4.5 mils per year.

Protective hydride films are black, vary widely in the degree of protection they offer, and appear to be highly sensitive to surface preparation. Chemically polished surfaces produce porous hydride films which offer little protection whereas electrolytically polished specimens produce dense films which are very resistant to hydrochloric acid.

The various mechanisms of corrosion are described and discussed in the light of the work of other investigators. Photomicrographs of the surface of various characteristic types of film formation are shown and their appearance is explained in terms of the postulated corrosion mechanisms. 6.3.20

Introduction

THE EXTENSIVE studies conducted by Lex B. Golden et al.[1,2,3,4,5] clearly reveal the diversified and outstanding corrosion resistance of zirconium. The zirconium metal used in the greater part of their work had been induction melted in graphite crucibles and contained carbon as a major impurity in amounts of 0.142 to 0.207 percent.[1] Golden showed that such amounts of carbon could be deleterious as well as beneficial in certain corrosive media. Since induction melting in graphite crucibles has been largely superseded by consumable electrode melting in arc furnaces of the "cold crucible" type, it is important that corrosion data be reported for metal melted by this process. Furthermore, since it will be more economical to use the less costly hafnium containing commercial grade zirconium for corrosion applications, the corrosion properties of this material will be of greatest interest to corrosion and design engineers.

Rates and modes of corrosion attack obtained in the laboratory are seldom, if ever, duplicated in the field. Nonetheless, the results are valuable as a rough, but necessary indication of the performance of the metal in environments approximating the experimental conditions or as a guide in the selection of materials for field tests.

The purpose of this paper is to make available basic and reference data on the corrosion resistance of commercial grade, consumable-electrode arc-melted zirconium in aqueous solutions of hydrochloric acid of standard chemical purity.

About the Author

W. E. KUHN is Supervising Engineer directing activities of Metallurgy Department, Research and Development Div., The Carborundum Company, Niagara Falls, N. Y. His activities include research and development in connection with zirconium, the high intensity arc, and more recently electric furnace practice. He was employed formerly by The Titanium Alloy Division, National Lead Co., Niagara Falls, N. Y. At Ontario Research Foundation, Toronto, Ontario he worked on powder metallurgy techniques applicable to titanium. He received the Young Author's Prize of the Electrochemical Society in 1953 for his papers on arc melting of reactive metals. Mr. Kuhn has a BSc in metallurgical engineering from University of Toronto and is a member of numerous engineering organizations, including NACE.

Test Procedures and Materials

Test Procedures

In general, solution concentrations and chemical additions were analyzed before and after each test. The surface appearance was noted and surface corrosion products were analyzed by X-ray diffraction. All specimens were examined beneath the microscope. The corrosion rate was measured by weighing the specimens before and after the test. Weighings were made to the nearest 0.1 mg. Duplicate tests were run in separate flasks for each corrosive environment.

The dimensions of the specimens are standard, being ½ inch x 2 inches x .045 inch. The specimens are surface finished to remove at least 0.001 inch with 120 grit silicon carbide abrasive cloth. The surfaces of the specimens were not activated by chemical treatment for removal of surface films prior to immersion in the test fluid.

The duration of the test is normally 144 hours (6 days) except where otherwise noted. Specimens are tested individually in wide mouth 1-liter Erlenmeyer flasks containing 600 cc of solution held at the test temperature in a constant water bath.

The following routine test procedures have been used in the present studies:

1. Aerated Aqueous Solution Tests at Temperatures of 35 C and 60 C of 144 Hours Duration

The apparatus and procedure used in tests conducted at temperatures below 100 C were in accordance with ASTM recommendations for "Tentative Method of Total Immersion Corrosion Test of Non-Ferrous Metals," ASTM Designation B185-43T.

The test flask design and general procedure employed in these tests may be found in reference 2. A photograph of the test apparatus is shown in Figure 1. Aeration of the solutions in each flask was individually controlled at 0.5 cubic feet per hour.

2. Boiling Solution Tests

a. *144 hour test.* The apparatus and procedure employed in the initial tests conform essentially to the "Boiling Nitric Acid Test for Corrosion-Resistant Steels," ASTM Designation A262-52T.

It was found, however, that the cold-finger condensers recommended in the above specification resulted in either concentration or dilution of the solution due to evaporation of either water or the acid for solutions other than the azeotropic composition. Allihn Pyrex reflux condensers with ground glass connections to the flasks were used with the solutions which presented this difficulty. This arrangement did not overcome entirely the problem of solution loss for tests of 144 hour duration. However, the concentration changes experienced were of little practical significance in the majority of tests.

b. *Long duration test.* This test was used to determine the changes in corro-

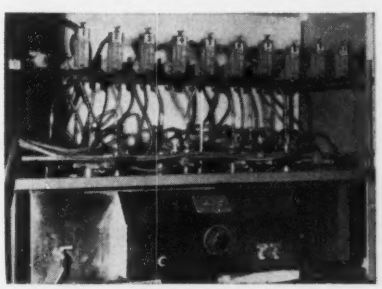

Figure 1—Apparatus used for corrosion tests conducted at 35 C and 60 C in aerated solutions.

★ Submitted for publication March 3, 1958. A paper presented at the Fourteenth Annual Conference, National Association of Corrosion Engineers, San Francisco, California, March 17-21, 1958.

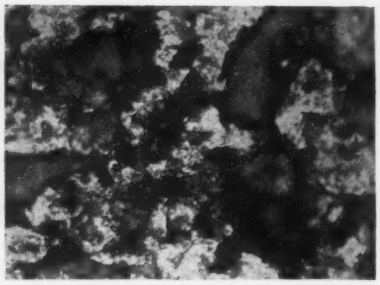

Figure 2—"As received" surface representative of Y (1), Y (2), and Y (3) zirconium. Cloudy areas represent pits in the surface. 500X reduced to 280X.

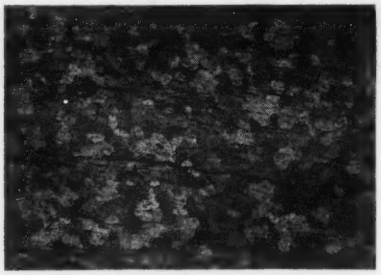

Figure 3—Photomicrograph representative of zirconium sheet fabricated from Y sponge showing striated zirconium hydride phase and the grain structure. Sample was chemically polished in 45 cc HNO_3, 45 cc H_2O, and 4-5 cc HF and stained in same solution diluted 1:4 with water. 350X reduced to 175X.

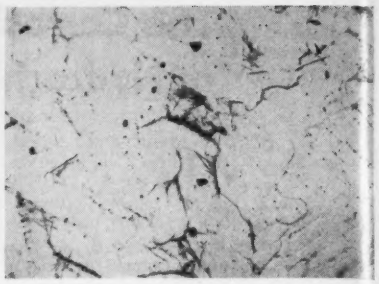

Figure 4—Photomicrograph of zirconium sheet having structure shown in Figure 3 after annealing at 1000 C for 5 hours in a purified argon atmosphere. Feathery precipitate is zirconium hydride phase which has precipitated out of solution on furnace cooling. Chemically polished. 200X reduced to 100X.

sion rate and mode of attack that took place over long periods of time. The test consisted of a series of 144 hour tests conducted in the equipment described above and continued for total elapsed times up to 2000 hours.

Materials

The corrosion resistance of several analyses of zirconium metal has been studied. Spectrographic and chemical analyses of the zirconium employed are given in Table 1.

Zirconium

Zirconium designated X represents hafnium-free zirconium sponge which has been single vacuum arc melted. The ingot was forged at 1450 F into a slab, pickled, and then hot rolled into strip at 1478 F using reductions of about 0.1 inch per pass. The hot-rolled strip was sandblasted and pickled to remove the oxidized surface layer.

Zirconium designated Y (1), Y (2), Y (3), represent three lots of commercial material double arc melted and fabricated into final sheet form by steel companies.

The "as received" surface of Y zirconium is represented by the photomicrograph in Figure 2. The cloudy areas in Figure 2 represent the locations of pits in the surface. It is believed that the pits were produced by a caustic pickle treatment.

The microstructure of Y zirconium is shown in Figure 3. The stringers are believed to be zirconium hydride phase formed during hot working operations and which subsequently became elongated during the cold rolling and level stretching operations. To confirm the presence of the hydride phase, the zirconium was annealed in purified argon for 5 hours at 1000 C to dissolve any hydride phase present and then furnace cooled to precipitate it from solid solution. The photomicrograph in Figure 4 reveals a feathery precipitate characteristic of the hydride phase formed by this treatment.

Corrosives

Commercial hydrochloric acid of standard chemical purity was selected for the present corrosion tests. The corrosion of zirconium in hydrochloric acid has been emphasized in the present corrosion studies because applications for zirconium in this acid are of current interest and show greater potential. The azeotropic concentration of hydrochloric acid (20 percent) having a boiling point of 107.5 C has been used in much of the work because of its severe corrosive properties and the constancy of the solution concentration when evaporation losses are encountered.

Results and Discussion

Short Term Tests

Relative Corrosion Resistance of Zirconium Metal of Different Chemical Analyses

The relative corrosion resistance of zirconium in boiling 20 percent hydrochloric acid is shown in Table 2. The weight changes varied widely from a gain in weight to a rate loss of less than 1 mil per year.

Although differences in impurity and hafnium content of zirconium may influence the corrosion resistance, any effect attributable to differences in impurity content of the zirconium probably has been masked by other factors influencing the corrosion rate.

Influence of Surface Preparations on the Short Term Corrosion Resistance of Zirconium

The influence of surface preparation on the corrosion rate in 20 percent boiling hydrochloric acid for 144 hour periods is shown in Table 3. Excellent corrosion resistance was shown in all instances. The corrosion rate was influenced by the character of the surface finish, the chemical and electrolytically polished samples exhibiting corrosion rates between 0.98 to 1.92 mils per year as against rates ranging from 0.05 to 0.13 mils per year for surfaces finished with 120 grit silicon carbide abrasive cloth. The variation in pit depths bears no apparent relation to surface finish or the corrosion rates in these specimens. The depth of pits originally present in Y material tested in the "as received" condition in hydrochloric acid showed no perceptible change with corrosion attack.

The Effect of Concentration and Temperature on the Corrosion Rate of Zirconium

As shown in Table 4, X zirconium exhibits excellent corrosion resistance in boiling solutions of hydrochloric acid up to 20 percent by weight. The corrosion resistance of X and Y zirconium in hydrochloric acid at 35 C and 60 C is also considered excellent.

Summary of Short Term Tests

The differences in corrosion rate produced by various surface treatments in the short term (144 hour) tests, suggested that the corrosion behavior of zirconium in hydrochloric acid is strongly influenced by the nature of the surface. Testing over extended times was undertaken therefore to explore more deeply the effect of surface condition on corrosion attack by hydrochloric acid and to explain the wide divergence in corrosion rates.

Long Duration Tests

The Corrosion Behavior of Electrolytically Polished and Vacuum Annealed Surfaces

Repeated 144-hour tests were continued for elapsed times of 2000 hours to obtain some insight into the mechanism of corrosion in boiling 20 percent hydrochloric acid. The results of an initial series of long duration tests for a series

TABLE 1—Chemical Composition of Zirconium Used in Corrosion Studies

Material Designation	Spectrographic and Chemical Analysis (Parts Per Million)												Knoop Hardness of Rolled Zr, 100 Gr. Load
	Al	Cr	Fe	Mg	Mn	Ni	Tb	Ti	V	Hf	N_2	C	
X	20	37	13	250	16	13	13	16	13	131	16.5	...	161.4
Y (1)	250	200	750	16	50	13	20	25	13	2.0 %	19.3	775	168.5
Y (2)	185	200	640	16	37	13	20	16	13	2.0 %	18.6	325	148.0
Y (3)	260	130	640	16	64	450	25	16	13	1.85%	...	200	185.0

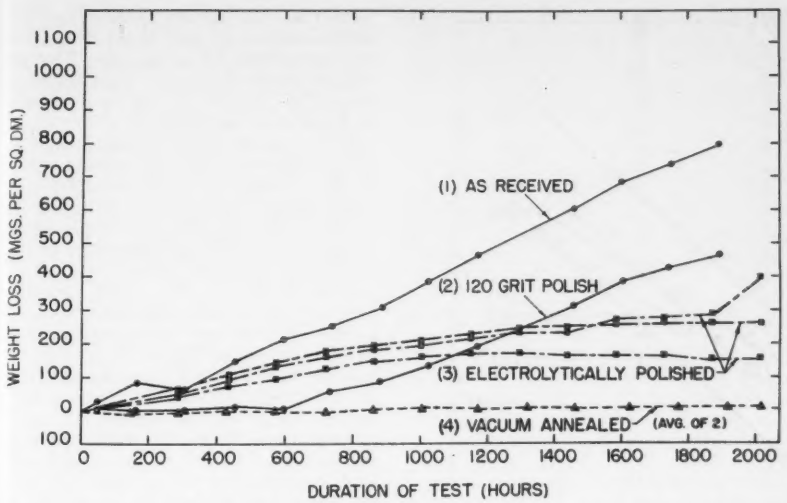

Figure 5—The influence of surface treatment on the corrosion behavior of zirconium in boiling 20 percent hydrochloric acid.

Curve	Surface Condition
(1)	"As received" (see Figures 2, 3, and 4).
(2)	Abraded with 120 grit SiC abrasive cloth.
(3)	Electrolytically polished in aqueous ammonium fluoride.
(4)	Abraded with 120 grit SiC abrasive cloth and vacuum annealed at 1100 C for 3 hours.

TABLE 2—The Relative Corrosion Resistance of Zirconium Metal of Different Chemical Analyses in Boiling 20 Percent Hydrochloric Acid

Material Designation	Corrosion Rate* (Milligrams Per Square Decimeter)	Mils Per Year	Pit Depth (microns)**	
			Average	Maximum
X	0.764	0.17	nil	nil
Y (1)	0.233	0.05	12	16
Y (2)	gain (0.3 mg)	gain	nil	nil
Y (3)	0.350	0.08	19	25

* Based on 144 hour test and surface finished with 120 grit silicon carbide abrasive cloth.
** Believed to be residual pits after surface preparation and produced originally by caustic pickling.

TABLE 3—The Influence of Surface Condition on the Corrosion Rate of Zirconium in Boiling 20 Percent Hydrochloric Acid

Material Designation	Description of Surface	Corrosion Rate*		Pit Depth*** (Microns)	
		Mgs Per Sq Dm Per Day	Mils Per Year	Avg	Max
X	Scratches produced by 120 grit abrasive belt grinder.	.764	0.17	nil	nil
X	Scratches produced by 3/0 polishing paper.	3.75	0.83	nil	nil
X	** Chemical polish	8.68	1.92	nil	nil
Y (1)	"As Received"	3.54	0.78	14	22
Y (2)	Scratches produced by 120 grit abrasive belt.	0.23	0.05	12	16
Y (1)	Scratches produced by 3/0 polishing paper.	0.64	0.14	14	20
Y (1)	** Chemical polish.	4.42	0.98	nil	nil
Y (2)	Electrolytic polish.	5.13	1.14	14	26
Y (2)	Vacuum annealed surface originally abraded with 120 grit abrasive belt.	1.45	0.320	21	26
Y (3)	"As received".	7.62	1.68	30	38
Y (3)	Scratches produced by 120 grit abrasive belt.	0.35	0.08	19	27
Y (3)	Scratches produced by 3/0 polishing paper.	0.70	0.15	17	27

* Based on 144 hour test.
** Chemical polish 45 cc HNO_3, 45 cc H_2O, 4-5 cc HF.
*** Believed to be residual pits after surface preparation and produced originally by caustic pickling.

of specimens subjected to a variety of surface treatments are given in Figure 5.

The surfaces of the specimens were rinsed thoroughly after each test, but care was taken to minimize the removal of corrosion products.

Three distinct sets of curves characteristic of the original surface preparation are evident. Specimens identified 1 and 2 in Figure 5 in the "as received" condition or abraded with 120 grit silicon carbide abrasive cloth exhibited the highest weight losses. It was also observed that after about 600 hours the corrosion rate of the specimen abraded with 120 grit abrasive cloth remained fairly constant until the termination of the test.

Specimens identified by curves 3 in Figure 5, that had been electrolytically polished in a saturated ammonium fluoride solution, exhibited initially high weight losses. After about 500 hours, however, the weight loss curves levelled off and even dropped slightly due to slight weight gains.

Vacuum annealing zirconium at 1150 C for 3 hours under a pressure less than 0.2 micron Hg resulted in remarkably low weight losses in boiling 20 percent hydrochloric acid after 2000 hours as shown by curves 4 in Figure 5.

The abraded specimen, curve 2, exhibited "breakaway" corrosion; that is, the initial corrosion attack was relatively insignificant up to a point when the specimens suddenly began to lose weight at a rate comparable to the "as received" specimen, curve 1, Figure 5.

In a special experiment, electrolytically polished specimens having approximately 0.01 to 0.03 mm of the original "as received" surface removed by abrasion exhibited maximum pit depths of 34 microns. These are believed to be residual pits after the abrasive polishing, which have been deepened and enlarged somewhat by the electrolytic polishing treatment. However, the removal of 0.07 mm of the original surface prior to electrolytic polishing resulted in the elimination of residual pits and a pit-free surface after electrolytic polishing. In general, the corrosion rate was not affected nor did pits appear to grow significantly in size or depth with time for any of the specimens represented in Figure 5.

Corroded electrolytically polished specimens, curve 3, Figure 5 displayed a thick, hard, black, adherent layer identified by X-ray diffraction as face centered cubic zirconium hydride with $a_o = 4.7738\text{Å} \pm 0.0018\text{Å}$. This corresponds to the compound ZrH. Microscopic examination of the surface of this layer revealed small rounded dimples containing a small amount of white sub-

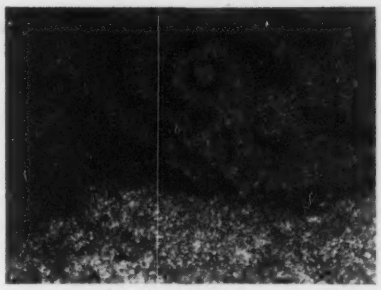

Figure 6—Layer of corrosion product on zirconium electrolytically polished in ammonium fluoride and exposed to boiling 20 percent hydrochloric acid for 2016 hours. Black hydride coating has been chipped away to reveal underlying metal surface at bottom of photomicrograph. Vertical illumination. 100X reduced to 50X.

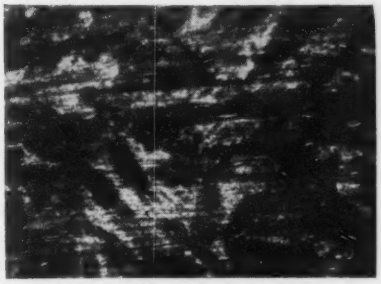

Figure 7—Appearance of surface of zirconium corrosion test coupon after exposure to boiling 20 percent hydrochloric acid for 2058 hours. Test coupon had been vacuum annealed for 3 hours at 1100 C prior to the test. Vertical illumination. 100X reduced to 50X.

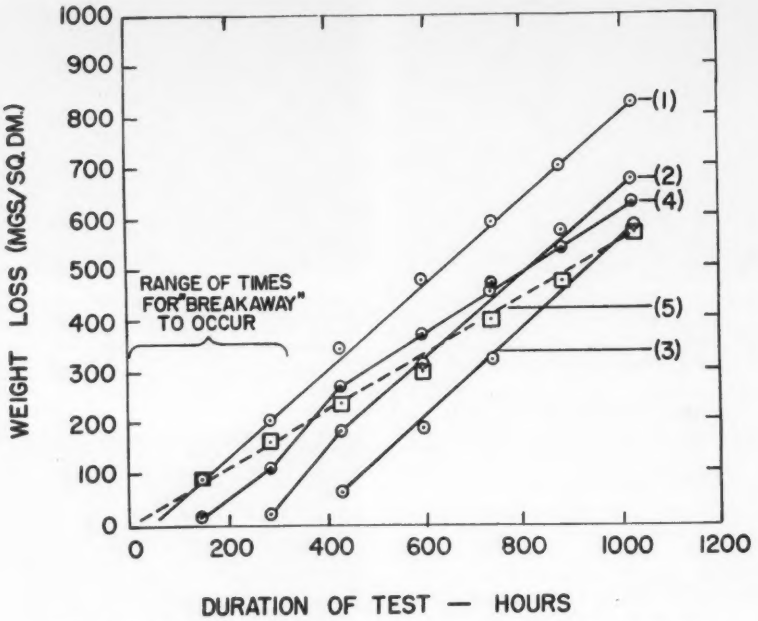

Figure 8—Effect of surface condition and vacuum annealing on the corrosion of zirconium.

Curve	Surface and Heat Treatment Prior to Corrosion Test	Type of Film
(1)	120X; VA 1100 C, 1 hr; CP	A (Figure 10, 11)
(2)	120X; CP VA 1100 C, 1 hr	A (Figure 10, 11)
(3)	4/0; CP VA 1100 C, 1 hr	A (Figure 10, 11)
(4)	120X; VA 1100 C, 1 hr; 120X	B (Figure 12, 13)
(5)	120X; CP	E (Figure 17)

120X = abraded with 120 grit SiC abrasive cloth.
4/0 = abraded with 4/0 metallographic polishing cloth.
VA = vacuum annealed at less than 0.1 micron Hg.
CP = chemically polished in a solution consisting of 45 cc water, 45 cc nitric acid, and 4-5 cc hydrofluoric acid. The original condition of the metal before surface and heat treatments is the "as received" condition for Y material shown in Figures 2 and 3.

TABLE 4—The Effect of Acid Concentration and Temperature on the Corrosion of Zirconium in Hydrochloric Acid

Material	Concentration of HCl (Weight Percent)	Corrosion Rate*		Temperature of Solution
		Mgs/dm²/dy	Mils/Year	
X	5	0.73	0.17	Boiling
X	10	0.33	0.07	Boiling
X	15	0.34	0.07	Boiling
X	20	0.76	0.17	Boiling
Y (2)	37	gain (0.35 mg)	gain	35C
Y (1)	20	0.29	0.06	35C
Y (1)	20	1.54	0.34	60C
X	20	2.25	0.50	60C

* Based on 144 hour test. Specimen surfaces prepared with 120 grit abrasive cloth.

stance. This was not identified, but is probably monoclinic zirconium oxide, a form of oxide commonly associated with hydride formation on the surface of zirconium. A portion of this black film was broken away to reveal the underlying metal surface roughened by small shallow pits as shown in Figure 6. The black layer is quite adherent and is removed with difficulty.

A dark coating, thin enough to reveal the underlying metal structure, was still present on vacuum annealed specimens after 2058 hours in boiling 20 percent hydrochloric acid as shown in Figure 7. A thin white film could be scraped or erased from the surface of this latter specimen. The black material was identified as zirconium monohydride and the white substance as monoclinic zirconium oxide.

The Corrosion Behavior of Vacuum Annealed Zirconium Subjected to Various Surface Treatments

Additional long duration tests were conducted to gain some insight of the corrosion mechanism and to confirm the passivity induced by vacuum annealing. These were conducted in boiling 20 percent hydrochloric acid for intervals of 144 hours for a total elapsed time of about 1008 hours. Vacuum annealed specimens were subjected to various surface treatments prior to and after vacuum annealing. Unannealed "as received" specimens were tested after abrading and chemically polishing their surfaces. After these tests, the surface films were thoroughly examined using X-ray diffraction and metallographic techniques. The results of these corrosion tests are summarized in Figure 8, and treatments 1 to 4 inclusive in Table 5.

It is apparent from the curves in Figure 8 that the corrosion mechanism is of the "breakaway" type whereby corrosion attack is relatively insignificant up to the transition point for a period of time determined by the durability of the primordial protective film.

After breakaway, the initial corrosion rate assumes a value anywhere between 2.9 and 4.5 mils per year as represented by the curves in Figure 8. A coating, henceforth designated Type A, and having the general appearance shown in Figure 10, forms on the surface. The rate of weight loss of material associated with A type films varied between 19.2 and 20.3 mgs/dm²/dy or 4.2 and 4.5 mils per year. The original surface of zirconium annealed at 1100 C for 1 hour exhibits the characteristic Widmanstätten structure of zirconium slowly cooled from above the transition temperature. Visually, the corroded surface presents a mottled pattern of elongated white patches against a dark background of powdery corrosion product. Under the microscope, the dark substance is seen to consist of a dark grey powdery corrosion product which has formed in layers of varying thickness as determined by the crystal orientation of the underlying crystal grains and delineated by the boundaries of both grains and Widmanstätten plates. Some films were very thin as evidenced by interference colors on certain grain faces.

The underlying metal has the appearance shown in Figure 11. Surfaces coated with the powdery grey material contained innumerable tiny corrosion pits. Surfaces that were bright and shiny were relatively free of pitting. Some individual grains were heavily coated while others were relatively free of the powdery grey material except for the occasional Widmanstätten plate running through the grain.

The sides of the corrosion deposit adjacent to areas free of corrosion product were quite steep and the corners remarkably sharp, indicating that the corrosion product is only slightly soluble in the acid.

X-ray diffraction of corrosion products similar to that shown in Figure 10 gave patterns of cubic zirconium hydride and very faint patterns of monoclinic zirconium dioxide.

A variation of the Type A film, designated herein Type B, was that produced by treatment 2 in Table 5 and represented in Figure 8 by curve 4. The significant difference between the surfaces of vacuum annealed specimens giving Type A and Type B films was that the former were chemically polished either before or after annealing and possessed smooth, relatively stress relieved surfaces, whereas the latter possessed a cold worked abraded surface prior to exposure to the acid.

The surface shown in Figure 12 consisted of a heavy layer of dark corrosion

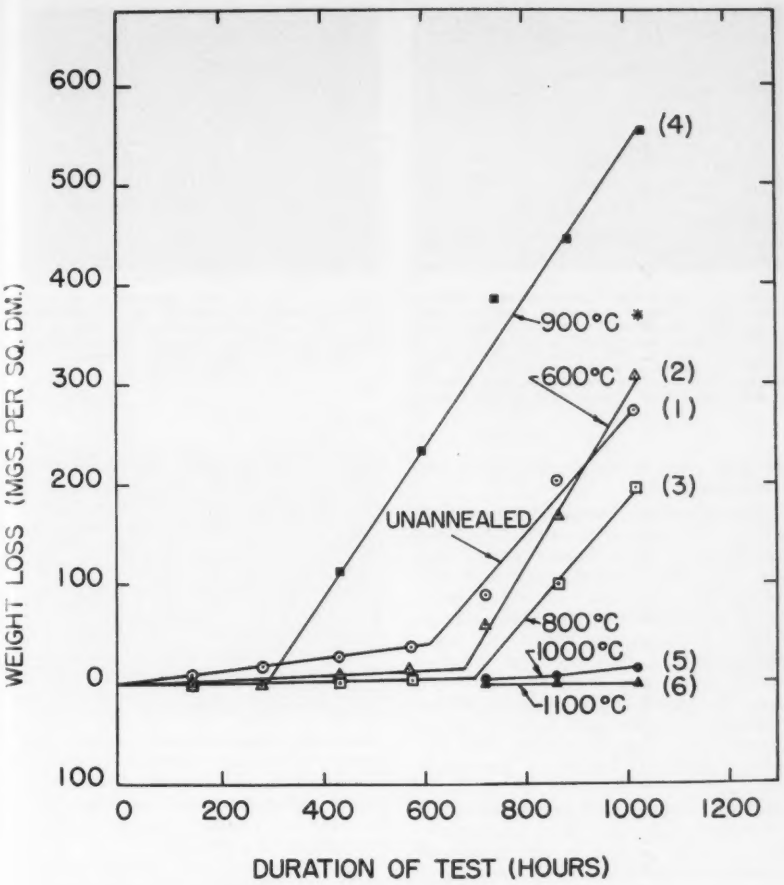

Figure 9—Effect of temperature of vacuum annealing on the corrosion of zirconium.

Curve	Surface and Heat Treatment Prior to Corrosion Test	Type of Film
(1)	120X	C (Figure 14)
(2)	120X; VA 600 C, 3 hours	C (Figure 14)
(3)	120X; VA 800 C, 3 hours	C (Figure 14)
(4*)	120X; VA 900 C, 3 hours	C (Figure 14)
(5)	120X; VA 1000 C, 3 hours	D (Figure 15)
(6)	120X; VA 1100 C, 3 hours	D (Figure 15)

* Data from a different series of vacuum annealing experiments.

TABLE 5—Summary of Observations of Nature of Films Formed on Zirconium After Corrosion Attack by Boiling 20 Percent Hydrochloric Acid

Surface and Heat* Treatment Prior to Corrosion Test	Approx Time to "Breakaway" (Hours)	Corrosion Rate After "Breakaway" (Mils Per Year)	X-Ray** Identification of Film Relative Intensity of Characteristic Pattern		Type of Film
			ZrH	ZrO_2	
1. 120X; VA 1100C, 1 hour; CP	<100	4.4	m	vf	A (Figure 10, 11)
2. 120X; VA 1100C, 1 hour; 120X	144	3.2	m	f	B (Figure 12, 13)
3. 120X; CP; VA 1100C, 1 hour	288	4.2	m/w	vf	A (Figure 10, 11)
4. 4/0; CP; VA 1100C, 1 hour	288	4.5	m	vf	A (Figure 10, 11)
5. 120X; VA 600C, 1 hour	576	4.5	w	m	C (Figure 14)
6. 120X; VA 800C, 1 hour	720	3.0	w	f	C (Figure 14)
7. 120X; VA 900C, 1 hour	288	3.9	s	w	C (Figure 14)
8. 120X; VA 1000C, 1 hour	>1008	nil	w	vf	D (Figure 15)
9. 120X; VA 1100C, 1 hour	>1008	nil	w	vf	D (Figure 15)
10. 120X	576	2.9	s	vf	C (Figure 14)
11. 120X; CP	nil	2.9	m/s	w	E (Figure 17)

* 120X—polished with 120 grit silicon carbide abrasive cloth, VA—vacuum annealed; CP—chemical polished in a solution made up from 45 cc water, 45 cc nitric acid, and 4 cc hydrofluoric acid.
** f = faint, w = weak, m = moderate, s = strong, v = very.

Figure 10—Type A Film: Structure showing Widmanstatten type structure of zirconium. The elongated light areas are uncoated whereas the dark areas are covered with a porous zirconium hydride film. Prior to corrosion, surface was abraded with 120 grit SiC abrasive cloth, chemical polished, and vacuum annealed at 1100 C for 1 hour. Surface appearance after exposure to boiling 20 percent hydrochloric acid for 1008 hours. Oblique illumination. 25X reduced to 12X.

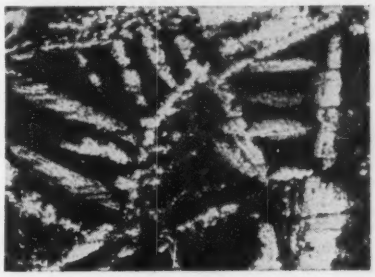

Figure 11—Type A Film: Underlying surface revealed by removal of hydride coating shown in Figure 10. Vertical illumination. 100X reduced to 50X.

product having an underlying Type A pattern. Overlying the Type A pattern of dark corrosion product was a thin, hard, white surface film. Contact between the black corrosion product and the white layer was interrupted where the white film bridged areas free of black corrosion product, thereby giving the surface a mottled appearance, similar to, but lighter in color than, the Type A surface film. The initial corrosion rate of this specimen was about 20 mgs/dm²/dy or 4.4 mils per year after breakaway, but changed to a lower rate of 14.4 mgs/dm²/dy or 3.2 mils per year after the third test period (432 hours) as shown in Figure 8, curve 4. The appearance of the underlying metal surface after removal of the corrosion product is shown in Figure 13 to be similar to that in Figure 11 for a Type A film.

Effect of Annealing Temperature

The influence of vacuum annealing temperatures between 600 C and 1100 C on the corrosion of zirconium are summarized in Figure 9 for surface treatments 5 to 11 inclusive, Table 5.

"Breakaway" corrosion was observed in all specimens annealed at 600 to 900 C inclusive, whereas it was not observed in specimens annealed at 1000 and 1100 C. There was a tendency for "breakaway" to occur earlier in specimens annealed at lower temperatures.

Specimens annealed at 600 C, 800 C, and 900 C exhibited surfaces having the appearance shown in Figure 14. This has been designated a Type C surface. The specimens annealed at 900 C were in-

Figure 12—Type B Film: Appearance of surface after exposure to boiling 20 percent hydrochloric acid for 1008 hours. Prior to corrosion, surface was abraded with 120 grit abrasive cloth, specimen vacuum annealed at 1100 C for 1 hour and the surface again abraded with 120 grit abrasive cloth. Oblique illumination. 25X reduced to 12X.

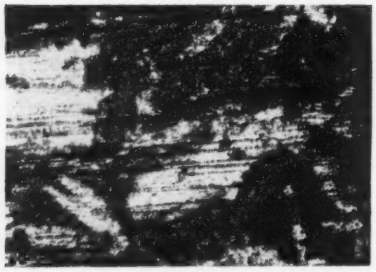

Figure 13—Type B Film: Underlying surface after removal of surface coating shown in Figure 12. Vertical illumination. 100X reduced to 50X.

cluded in an entirely different series of experiments. This is additional evidence of the unpredictable behavior of the primordial oxide films. The surface layers invariably consisted of a black powdery corrosion product underlying a loose, white, flaky, film. Since the white film was loosely adherent, those specimens having breakaway points closest to the termination of the test retained the larger amounts of white material. The distribution of the white material was uneven, some areas having lost more than others. The underlying metal surface suffered shallow pitting attack. The rate of weight loss varied between 13.2 and 20.4 mgs/dm²/dy or 2.9 and 4.5 mils per year. The X-ray patterns for monoclinic zirconium dioxide were generally stronger for Type C than Type A surfaces whereas the zirconium hydride patterns were weaker as would be expected assuming the white overlying surface film is monoclinic zirconium dioxide.

Annealing temperatures of 1000 C and 1100 C resulted in relatively insignificant corrosion attack after 1008 hours. The appearance of the surface after termination of the test is shown in Figure 15. This is referred to as a Type D surface. A mottled pattern similar to that in Type A and B surfaces appears to be developing on the specimen annealed at 1000 C and might be an indication of the initiation of "breakaway" corrosion. Probing of the surface of this specimen causes a thin white flaky film, otherwise transparent and undetectable, to be removed. The surface was dark in appearance indicating the presence of zirconium hydride. Both cubic zirconium hydride and monoclinic zirconium dioxide were identified by weak X-ray patterns in the corrosion product.

Figure 14—Type C Film: Surface showing overlying flakey, white film of monoclinic zirconium oxide (light areas) and underlying hydride coating (dark areas). Specimen was exposed to boiling 20 percent hydrochloric acid for 1008 hours. Prior to corrosion, surface of specimen was abraded with 120 grit abrasive cloth and the specimen vacuum annealed at 800 C for 1 hour. Oblique lighting. 25X reduced to 12X.

For comparison, unannealed specimens with a 120 grit surface finish identified by treatment 10 in Table 5 and in Figure 9 by curve 1 were included in this series. This specimen exhibits "breakaway" corrosion, a C type film, and suffered a corrosion rate loss of 13.2 mgs/dm²/dy or 2.9 mils per year. Shallow pitting attack and relatively unattacked areas elongated in the direction of the polishing scratches and normally not present in a Type C surface were evident as shown in Figure 16. The bright elongated areas shown in the photomicrograph were covered with a thin white protective oxide film. Some white material also was present in the grooves of scratches underlying which was a black corrosion product, believed to be hydride. It appeared that the patches of remaining white scale, though weakly adherent, protected the metal surface against corrosion.

The effect of subjecting the abraded surface of unannealed "as received" zirconium to a chemical polishing treatment is summarized by treatment 11 in Table 5 and by curve 5, Figure 8. A dark adherent film develops early in the test gradually becoming black as the test proceeds. This is referred to as a Type E film. A characteristic linear loss rate of 13.1 mdd or 2.88 mils per year was exhibited. The film, identified as cubic zirconium monohydride, is very smooth and free of pitting attack. The underlying metal surface, after erasing the film, is shown in Figure 17. Its surface has been roughened by a uniform distribution of innumerable shallow corrosion pits.

Mechanism of Corrosion in Hydrochloric Acid

Three characteristic modes of corrosion attack have been identified with zirconium in hydrochloric acid in these studies. They are:

A. "Breakaway" corrosion of abraded and vacuum annealed metal surfaces.

B. Corrosion of chemically polished surfaces having porous hydride surface films.

C. Corrosion of electrolytically polished specimens having dense, coherent, hydride surface films.

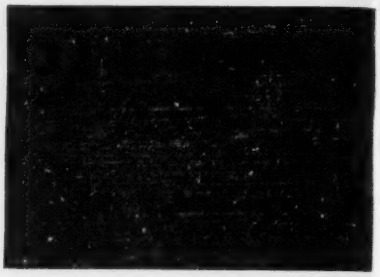

Figure 15—Type D Film: Appearance of surface after 1008 hours in boiling 20 percent hydrochloric acid. Prior to corrosion, surface was abraded with 120 grit abrasive cloth, specimen vacuum annealed at 1000 C for 1 hour. Oblique illumination. 25X reduced to 12X.

Breakway Corrosion

In this mode of attack, the initial slow rate of reaction at the metal surface is determined by the rate of diffusion of reactants through an oxide film. After an indeterminate period of time, there is a sudden increase in the rate of attack when "breakaway" corrosion occurs. The evidence suggests that this occurs when the oxide film disrupts thereby allowing the electrolyte relatively free access to the metal surface. Repeated identification of face-centered cubic zirconium monohydride with overlying films of monoclinic zirconium oxide indicated that hydride was being formed beneath the oxide film thereby destroying coherency between the metal and the protective film. This would lead to rapid loosening and cracking of the film and a sudden change to another mechanism of corrosion.

The process whereby hydrogen reaches the metal surface has not been satisfactorily explained. Hydrogen may diffuse through the oxide barrier as hydrogen ions or molecules through cracks, grain boundaries and other structural defects. That hydrogen diffuses through this film is reasonably certain. Gulbransen and Andrew have shown that oxide films produced by an oxygen atmosphere have a remarkable influence on the rate of reaction of hydrogen with zirconium.[6] Furthermore, they indicated that the character of the film, rather than its thickness was of even greater significance.

Studies indicate that the zirconium oxide film possesses an anion defect lattice and is a N-type semiconductor. Film growth in oxygen and aqueous environments is believed to take place by the inward diffusion of oxygen ions with the formation of oxide at the metal-oxide interface.[7,8,9] The surface oxide film identified with the corrosion of zirconium in this work was found to be monoclinic ZrO_2. The rate determining process for such a lattice is thought to be a repetitive exchange process whereby anion holes $h(O^{-2})$ and quasi-free electrons (Zr^{+++} ions) diffuse to the metal outer surface and oxygen ions diffuse inward to the metal oxide interface where they react to form oxide and produce oxygen-ion vacancies and quasi-free electrons.

It is not known how the anion defect character of the oxide film influences the protective qualities of the film in aqueous media with respect to the migration

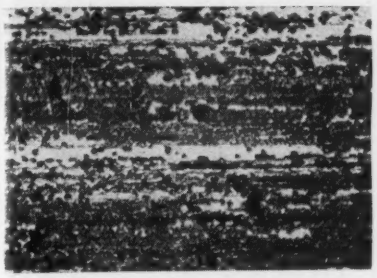

Figure 16—Type C Film: Appearance of underlying surface after 1008 hours in boiling 20 percent hydrochloric acid. Prior to corrosion, surface was abraded with 120 grit SiC abrasive cloth. White areas are at the bottom of grooves produced by abrasion and were covered with a flakey zirconium oxide film before removal with a soft erasure. Vertical illumination. 100X reduced to 50X.

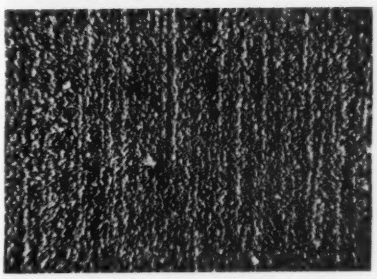

Figure 17—Type E Film: Underlying metal surface after removal of black cubic zirconium hydride coating. Prior to corrosion, surface was first abraded with 120 grit SiC abrasive cloth and then chemical polished. Oblique illumination. 25X reduced to 12X.

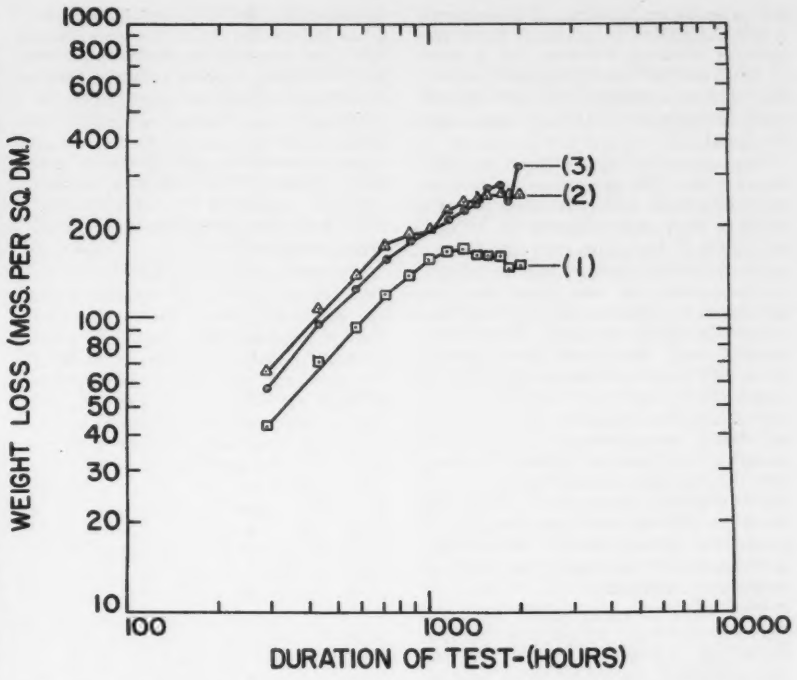

Figure 18—Weight changes of electrolytically polished specimens corroded in boiling 20 percent hydrochloric acid.

Curve	*Value of Constants C and n in Relation* $W = Ct^n$ *Up to the Transition Point*	
(1)	0.081	1.11
(2)	0.165	1.06
(3)	0.113	1.10

of hydrogen and oxygen ions. Three mechanisms considered to play a dominant role in aqueous solutions of hydrochloric acid are:

1. Formation of an extremely thin primordial passive film which is amorphous, continuous, and impervious to the diffusing ions. The existing theories of passive films may possibly explain the nature of this film. Discontinuities are believed to develop through the gradual transformation of the amorphous film into a crystalline film containing the usual defects associated with a crystalline lattice.

The conditions of formation for such films are probably critical; hence, consistent and uniform reproduction of the amorphous type film on a zirconium surface might be difficult to achieve in practice.

2. Oxide film growth by anodic migration of oxygen ions through the already established oxide lattice under the influence of the low strength electric field of the electrolyte, followed by breakdown of this film after a certain thickness as a result of stresses produced by the mismatch between the oxide and metal lattices. Defects in the metal surface such as hydride inclusions may be a factor in eventual failure of this film.

3. Migration of hydrogen through the anion defect oxide lattice to form hydride at the metal-oxide surface. It is known that the rate of migration of hydrogen is related to the character of the film as already indicated, but it is not known whether it is specificially related to the anion-defect character of the film. It has been suggested[10] that hydrogen ions combined with oxide ions in the oxide lattice may migrate to the metal surface by a transfer mechanism. They may also be interstitial with no attachment to any oxide ion. Hydrated oxide may be another source of hydrogen ions in zirconium oxide although this is considered unlikely. It has also been shown that a high initial hydrogen content of the metal accelerates the rate of absorption of hydrogen.[11]

In water or aqueous solutions, oxygen-ion vacancies and quasi-free electrons in the oxide lattice move by an anodic process from the metal-oxide interface to the outer surface through the oxide. The anion holes react with water at the surface producing oxygen ions in the outer layer of the oxide lattice and at the same time release hydrogen ions in accordance with the equation:

$$h(O^{-2}) + 2e + H_2O \rightarrow O^{-2} + 2H^+$$

The rate of migration of oxygen ions will be dependent on the oxygen-ion vacancy sites available at the surface and interior of the oxide. At low temperatures, less than 150 C, and in water this reaction is negligible. In electrolytes such as boiling aqueous hydrochloric acid solutions at ambient atmospheric pressures, the chemical potential across the film may be high enough to appreciably influence the rate of migration of oxygen ions through an intact, coherent, oxide film. Chlorine ions in an anodizing cell are reported to have no effect on the anodic process below the impressed corrosion voltage.[12] This suggests that the relatively large chlorine ion *per se* probably has little effect on diffusion of oxygen ions through the oxide lattice.

Regardless of the precise manner in which the above mechanisms operate, the character of the oxide film formed on zirconium undoubtedly determines its protective qualities.

The concentration of defects in oxide films formed by vacuum annealing is probably highly sensitive to temperature, pressure, time, and the original state of the surface. The cooling of zirconium in high vacuum from a temperature that would dissolve any surface oxide film would be most critical since the leak rate of the system and the time to cool largely determines the thickness and the degree of perfection of the oxide lattice. It has been found, for example, that the corrosion resistance of zirconium in hot water is highly dependent on heat treatment.[13]

It may be shown, by an analytical treatment analogous to one given by Wagner, that the concentration of oxygen-ion vacancy sites at the outer surface of the oxide increases rapidly with decreasing partial pressure of oxygen at a given temperature.[14] Therefore, depending on the conditions of its original formation, an oxide film may vary widely in the concentration of oxygen ion vacancies and the steepness of the concentration gradients across the film thickness.

The experimental results have shown

the pronounced influence of vacuum annealing. Table 6 summarizes the conditions of vacuum sintering for a series of experiments. No correlation between the vacuum sintering conditions and the wide variation in breakaway times could be found.

Experiments 3 to 7 in Table 6 clearly indicate that the annealing temperature is an important factor. Annealing at and cooling from temperatures of 1000 C and 1100 C has apparently produced a protective film approaching the desired characteristics. It was noted that the specimens in experiments 3 to 7 in Table 6 were decidedly tarnished. Those specimens having breakaway times greater than 1000 hours and annealed at 1000 C and 1100 C displayed a metallic, though dull sheen after annealing; those annealed at 1100 C in experiment 2 and having breakaway time of about 370 hours exhibited a high metallic lustre with no visible evidence of an oxide film. This lends support to the premise that an oxide film having special characteristics is essential for improved corrosion resistance of zirconium.

The desired oxide films are most likely to be formed on surfaces that have been first treated to remove surface contamination, imperfect films, and hydride phases. One effective treatment is annealing in high vacuum above the recrystallization temperature and at a temperature which removes both gaseous and volatile impurities as well as oxide or other films already present. Recrystallization is beneficial if credence is given to the principle of variable occlusive capacity.[15] This theory states in effect that the hydrogen concentration of metal in the strained or plastically deformed condition, such as an abraded surface, may be many times the solubility of hydrogen in fully annealed metal. Recrystallization or annealing is thought to close the rifts and voids in the metal structure, thereby minimizing the amount of hydrogen occluded in the metal surface and lattice which presumably would be available for hydride formation.

Thus the structure and thickness of the original oxide film prior to immersion in the electrolyte is thought to be a significant factor determining the time for "breakaway" corrosion to occur.

After "breakaway," corrosion is by chemical dissolution as expressed by the over-all reaction.

$$Zr + 2HCl + H_2O \rightarrow ZrOCl_2 + 2H_2$$

The zirconium tetravalent ion has a high ionic potential and does not exist as a simple dissolved ion but rather as hydrated zirconium oxide or oxychloride. Part of the hydrogen evolved in the reaction may combine with zirconium as zirconium hydride at cathodic areas forming various types of black or dark grey porous coatings on the metal surface as already described.

Oxygen is believed to be a necessary part of the above mechanism, since one oxygen ion and two chlorine ions must be present at a reaction site to provide sufficient energy to cause the release of a zirconium ion from the metallic lattice. The oxychloride molecule immediately hydrates, forming colloidal particles of hydrated zirconium oxychloride. It is suggested that during corrosion, any oxygen in the metal lattice remains tightly bonded to the zirconium atoms. The presence of monoclinic zirconium oxide is explained by the oxidation of hydride in the porous zirconium hydride surface coating.

Another possible explanation for the presence of part of the hydride beneath the protective film is the in situ formation of hydride from hydrogen already present in the metal lattice. It is conceivable that removal of zirconium ions through reaction with oxygen ions at the metal-oxide interface might concentrate hydrogen at the metal surface as hydride, but not in the amounts observed.

Hydride inclusions may be responsible for initial stages of breakdown of the oxide film through the action of hydrogen, released by the dissolution of zirconium hydride, exerting pressure on the oxide film in the region of the inclusion. Relief of localized stresses induced in the metal by such inclusions may also be a factor in the formation of cracks and other structural defects in the oxide film.

At pressures less than 0.1 micron Hg and initial hydrogen contents between 28 and 67 parts per million, temperatures in excess of 800 C are required to reduce the hydrogen content to 6 or 12 parts per million.[16] This is considered an additional benefit of vacuum annealing at temperatures above 800 C.

Corrosion of Chemically Polished Surfaces

The effect of removing surface films chemically in a H_2O-HNO_3-HF solution on the mode of surface attack and corrosion rate of unannealed and annealed specimens is represented by curves 5 and 1 respectively in Figure 8.

Corrosion attack of unannealed zirconium having the structure shown in Figure 3 is characterized by the formation of black, face-centered cubic zirconium hydride film and a linear corrosion rate of 13.1 mdd or 2.9 mils per year. The film is quite porous, which explains the linear corrosion rate.

The corrosion attack of zirconium vacuum annealed at 1100 C for 1 hour and having the typical basket weave structure, is characterized by a mottled, dark grey cubic zirconium monohydride film as shown in Figures 10 and 11 and a linear corrosion rate of 20 mgs/sq dm/day or 4.4 mils per year. This film was also very porous giving a linear corrosion rate.

The significant difference between the unannealed and annealed specimens is that a continuous porous film was formed on the former and a patchwork porous film on the latter due to grain orientation effects. The porous hydride film reduces corrosion attack by slowing diffusion of the corrosive solution to the metal surface. Since only a portion of the surface of the annealed specimen is shielded in this way as revealed in Figure 10, a higher corrosion rate would be expected.

Unannealed specimens were polished by immersing in a chemical polishing solution consisting of 45 cc water, 45 cc nitric acid and 4 cc hydrofluoric acid. They were rinsed thoroughly, and then immersed in the boiling 20 percent hydrochloric acid immediately, rather than

TABLE 6—Summary of the Influence of Vacuum Annealing* on the Corrosion of Zirconium

Vacuum Annealing Experiment	Annealing Temp, DegreesC	Time at Temp, Hours	Vacuum at Annealing Temp, Microns	Vacuum at Start of Cooling Cycle, Microns	Leak Rate, Microns Per Min	Furnace Vacuum at End of Cooling Cycle, Microns	Appearance of Surface Specimen After Annealing	Approx. Time Before Breakaway, Hours
1.....	1100	3	.18-.06	.06	.4	.01	Very shiny, metallic lustre	>2000
2.....	1100	1	.12-.07	.07	.33	.01	Bright shiny metallic lustre	~370
3.....	600	1	.1 -.18	.18	.3	.01	Tarnished (straw yellow)	~680
4.....	800	1	.07-.03	.03	.3	.01	Tarnished uniform straw yellow	~700
5.....	900	1	.12-.07	.07	.4	.01	Bright shiny metallic lustre	<288
6.....	1000	1	.05-.03	.03	..	.01	Bright metallic, portion a dull gray due to oxidation	>1008
7.....	1100	1	.07-.06	.06	..	.01	Metallic gray white with a dull sheen	>1008

*Vacuum Annealing Conditions: Specimens suspended by a molybdenum wire in a zirconium lined graphite container in evacuated furnace tube.

after a delay of hours or days; these specimens produced an average corrosion rate of 31.4 mgs/dm²/dy or 7.0 mils per year for an exposure time of 144 hours.

These observations suggest that the formation of the hydride coating is not only dependent on grain orientation but is also highly sensitive to the effect of films formed immediately prior to immersion in the hydrochloric acid. A few isolated patches of white substance identified as monoclinic zirconium oxide had formed on the chemically polished surface, thereby indicating that oxide films may form after immersion in the hydrochloric acid.

Corrosion of Electrolytically Polished Surfaces

The logarithmic graph of the unit weight loss versus time in Figure 18 yields a straight line for specimens electrolytically polished in a saturated ammonium fluoride solution up to about 720 hours, beyond which the corrosion weight loss begins to decrease or to change to a weight gain.

The corrosion reaction for the straight line portion of the curves in Figure 18 may be represented by an empirical equation of the form:

$$W = Ct^n$$

or

$$\log W = n \log t + \log C$$

Where W is the unit weight loss after time t, n is the slope of the curve, and C a rate constant.

When n is equal to unity the rate law is linear. When n is less than unity the instantaneous rate of corrosion is a decreasing function and when greater than n, an increasing function. The experimental values for n in this relation for curves 1, 2, and 3 representing individual specimens are 1.11, 1.06 and 1.10 respectively and for the rate constant C are 0.081, 0.165, and 0.113 respectively. The differences between the rate constants are believed to reflect variations in the permeability or diffusivity of the hydride film.

Although a satisfactory explanation for the transition to lower corrosion rates after 720 hours cannot be presented, it is believed to be related to changing density, and hence the permeability of the hydride layer. There is evidence, for example, that when hydrogen evolution is slow at the surface of zirconium, black, hard, dense films are formed, whereas at more rapid rates of hydrogen evolution the films tend to be powdery.[17]

Adsorption of hydrogen in the zirconium lattice may also be a factor in any explanation of the corrosion characteristics displayed in Figure 18. The transition metals in general exhibit passivity which is believed to be closely related to the partially filled d-electron band. Filling the d-band with electrons from hydrogen may have an important influence on the corrosion of zirconium as it has on other metals and alloys such as the iron-chromium alloys, in which the potentials of alloys cathodically charged with hydrogen are shifted to more active values.[18]

Due to the relatively large difference in the densities of zirconium hydride and zirconium metal, the brittle hydride film may be expected to crack or separate from the metal after a critical thickness has been attained, thereby possibly explanning the sudden losses in weight in two of the curves in Figure 18.

TABLE 7—Summary of Characteristics of ZrH Films Formed on Zirconium

History of Metal Surface Prior to Corrosion Test in Boiling 20% HCl	Description of Zirconium Hydride Film after Corrosion Test	Post Transition Corrosion Rate	
		Mgs/dm²/day	Mils Per Yr.
1. As received zirconium sheet having structure shown in Figure 3, surface finished with 120 grit SiC abrasive cloth and electrolytically polished in saturated ammonium fluoride solution.	Continuous, adherent, hard, black film of zirconium monohydride. Corrosion obeys logarithmic rate law during first 720 hours after which corrosion rate suddenly decreases. (see Figure 18)	First 720 hours assuming linear rate law	
		4.0 to 6.8	0.9 to 1.3
		Average for 2000 hrs. assuming linear rate law.	
		1.8 to 3.9	0.4 to 0.9
2. As received zirconium sheet having structure shown in Figure 3, surface finished with 120 grit SiC abrasive cloth, chemical polished, and exposed to air prior to testing.	Continuous black, powdery film of zirconium monohydride. Corrosion obeys linear law.	13.1	2.9
3. As received zirconium sheet having structure shown in Figure 3, surface finished with 120 grit SiC abrasive cloth, fully activated by chemical polishing and immediately immersed and tested in boiling acid.	Grayish white metallic matte surface free of oxide or hydride films.	31.4	7.0
4. As received zirconium sheet having structure shown in Figure 3, surface finished with 120 grit SiC abrasive cloth, vacuum annealed at 1100C for 1 hour, and chemical polished. Bright annealed surface free of visible films and exhibiting typical basketweave structure of annealed zirconium metal.	Patchwork pattern. Approximately 80 percent of area covered with black powdery zirconium monohydride. Remaining area free of surface film. Hydride is formed on areas delineated by the boundaries of grains and Widmanstatten plates. Corrosion obeys linear rate law (see Figure 10, 11)	20.0	4.4
5. As received zirconium sheet having structure shown in Figure 3, surface finished with 120 grit SiC abrasive, cloth, chemical polished, vacuum annealed at 1100C 1 hour. Bright annealed surface free of visible films and exhibiting typical basket-weave structure of annealed zirconium.	Same as described in 4 above (See Figure 10 and 11)	19.2	4.2

Discussion and Summary

Accumulating evidence clearly indicates that optimum corrosion properties in zirconium for nuclear and chemical applications depends on the specific properties of a primordial protective zirconium oxide film. The desired characteristics of this film have not been described in detail, nor can treatments be developed and prescribed for their attainment until these details are known.

In general, the search has been narrowed to a very thin film possessing properties which minimize the migration of oxygen and/or hydrogen to the metal-oxide interface. Failure of primordial oxide films is believed to occur by stress cracking of films that have grown beyond a critical thickness, by the formation of a hydride layer of critical concentration which causes loosening and flaking away of the oxide film, or by a combination of these mechanisms.

The fact that films have been encountered which effectively resist corrosion in acid environments for thousand of hours as against others lasting but a few hundred hours or less, suggests that a film of specific properties is involved. It is imperative, therefore, that the desired films be produced, isolated, and their growth characteristics and properties studied. The anion defect character of monoclinic zirconium oxide films is thought to have an important bearing on this problem—hence studies of the semiconductor electrical properties, correlated with the corrosion properties of the films, should yield interesting data.

The formation of hydride films has been found to play an important role in the corrosion of zirconium in boiling 20 percent hydrochloric acid. Zirconium hydride films afford limited protection through their ability to hinder the diffusion process, and hence the corrosion reaction, at the metal surface. The degree of protection afforded varies widely depending upon the density and the permeability of the hydride film. The properties of the hydride film are in turn influenced by the nature of the surfaces upon which they are formed. The properties of a film may be modified during its growth, thereby affecting changes in the corrosion rate. A summary of the various types of hydride films encountered in this study is given in Table 7.

Table 7 shows that the protective hydride film produced by electrolytic polishing is superior to other surface treatments. Development of a well defined grain structure appears to have impaired the corrosion resistance of zirconium by the exposure of crystal faces to the corrosive environment which do not form protective zirconium hydride films.

The corrosion rate of the annealed zirconium is intermediate between the corrosion rates for surfaces of unannealed metal having continuous hydride films and those with fully activated film-free surfaces; namely 2.9 and 7.0 mils per year. The resultant corrosion rate for the annealed metal may be expected to depend on the relative proportions of

hydride coated and film-free surface and the characteristic corrosion rates for these surfaces. The characteristic patchwork pattern of hydride coated crystal faces is displayed whether the vacuum annealed surface is untreated, chemical polished, or abraded. Vacuum annealing therefore appears to be advantageous only when an impervious, adherent primordial zirconium oxide film of long life is formed.

The hydride films underlying protective monoclinic ZrO_2 films are porous and obey a linear rate law with values of the rate instant ranging between 2.9 and 4.5 mils per year.

Corrosion rate values for zirconium in hydrochloric acid obtained from short term tests are misleading since they do not provide information on the length of time before "breakaway" occurs or the post transition corrosion rate. The desired information is best provided by a series of short term tests using the original specimens throughout the test for a total elapsed time sufficient to establish the "breakaway" point and the post transition corrosion rate.

The approximate average corrosion rate where corrosion is of the "breakaway" type is given by the expression:

$$R = \frac{(T_2 - T_1)k}{T_2}$$

where R is the average lineal corrosion rate, T_1 is the time to the "breakaway" point, T_2 is the total time of the test, and k is the post transition corrosion rate. It is assumed that the corrosion rate prior to breakdown of the zirconium oxide film is negligible.

Since the post transition rate seldom exceeds 20.4 mgs/dm²/day or 4.5 mils per year in boiling 20 percent hydrochloric acid, this may be considered a maximum value for temperatures up to the boiling point (107.5 C) and HCl concentrations up to 20 percent hydrochloric acid. In general, corrosion rates are likely to be less than 4.5 mils per year since oxide films are normally present which decrease the effective corrosion time $(T_2 - T_1)$ and because most hydride films have rate constants less than 4.5 mils per year.

Although corrosion rates less than 5 mils per year are considered excellent for hydrochloric acid environments, the corrosion resistance of zirconium may possibly be further enhanced by directing research to improvement in the length of time for "breakaway" to occur and by minimizing the post transition corrosion rate. The development of surface treatments which produce impervious hydride films on the surface of zirconium is suggested as another method for improving the corrosion resistance of zirconium in hydrochloric acid.

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DISCUSSION

Comment by Byron B. Burd, Damascus Tube Company, Greenville, Pa.:

In answer to the questions "What corrosion effects have you observed from nitrogen" and "What purity can be maintained in welds" we replied as follows.

Our experience has been that where proper shielding has been employed, gaseous contamination has been well below the established limits. Nitrogen, hydrogen and oxygen pickup is controlled by using specially designed trailing gas shields on both the OD and ID of zirconium, zircaloy, titanium and tantalum tubing. The trailing shields utilize a mixture of argon and helium fed from a master mixing cylinder. Gas flow should be controlled to obtain a great volume of flow at low pressure. Too much pressure causes a swirling action to take place which in turn invites the inspiration of air.

Welds which were protected by shielding have been found to be of similar purity and ductility as the base metal upon chemical and metallurgical examination.

Question by Cornelius Groot, Richland, Washington:

What is the mg/dm² per mil loss for zirconium?

Reply by W. E. Kuhn:

The per mil loss of surface metal is 1648 mg/dm².

Comment by George J. Puckett, The Dow Chemical Company, Pittsburg, California:

Our experience with W/Zr in fused NaOH and KOH at 700 F aerated is as follows: We exposed one Zr coupon and found the rate comparable to that for Ni (about 20 mils/yr). Our environment does corrode Ni in some areas.

Question by Alan G. Caterson, Crucible Steel Company, Titanium Division, Midland, Pennsylvania:

Is any difference in corrosion rate noted between weld metal, heat affected zone and base metal in Zr welds exposed in HCl?

Reply by W. E. Kuhn:

Zirconium sheathed immersion heaters having caps welded by the tungsten inert gas process show no evidence of preferred corrosion attack in welded areas of the heaters after exposure to boiling film heat transfer conditions in 20 percent hydrochloric acid for periods up to 500 days. These tests are still underway with little evidence of corrosion other than a thin scale of mixed zirconium hydride and monoclinic zirconium dioxide.

Question by R. McFarland, Hills McCanna Co., Chicago, Illinois:

Does the presence of hafnium as an impurity affect and increase corrosion rate? Also what is the effect of ferrous and ferric ions on increased corrosion rate?

Reply by W. E. Kuhn:

Hafnium has no perceptible effect on the corrosion rate in boiling 20 percent hydrochloric acid at temperatures up to the boiling point. The effect of hafnium content at higher temperatures was not investigated.

Zirconium is sensitive to the presence of iron in hydrochloric acid solutions, increased corrosion rates being observed at the higher iron concentrations. This is shown by the data in Table D-1.

TABLE D-1—Corrosion of Commercial Zirconium in Boiling 20 Percent HCl Containing Iron Impurity (6 Day Test)

Iron Concentration (parts per million)	Mils Per Year
5	0.3
10	0.5
50	2
60	2
130	4
500	12
1000	22

Long duration tests up to 1008 hours in boiling 20 percent hydrochloric acid containing iron in amounts up to 1000 ppm exhibited increasing pitting attack with iron concentration; however, damage was slight at concentrations below 10 ppm of iron. Corrosion in commercial muriatic acid was found to be the same as in acid of standard chemical purity.

Continuing tests in 20 percent hydrochloric acid containing iron up to 1000 parts per million indicate that zirconium is highly resistant to attack at a boiling film surface after eleven months exposure, although shallow pitting attack is evident.

Corrosion in the Corn Wet Milling Industry*

By RICHARD BENES, FRED J. HOLSINGER and RUSSELL E. PIERSON

Abstract

Use of dilute sulfurous acid for steeping corn prior to milling results in numerous corrosion problems. Corn syrup, dextrose, corn oil, and special starch production using such raw materials as sulfuric acid, hydrochloric acid and sodium hypochlorite make it extremely important that the proper construction material is selected. The occurrence of stress cracking of evaporator tubes and selection of materials for various processes are discussed briefly. The use of stainless steel, sheet linings and specialized organic finishes have proved effective in combating the more serious corrosion and abrasion problems. 8.3.5

Introduction

THE CORN wet milling industry in the United States dates back to 1848. Since that time, the industry has risen to a major role in food and consumer product manufacturing. Important products from corn include starch, corn syrup, and corn oil. Dozens of industrial applications, ranging from the production of antibiotics to the purification of aluminum ore, consume large quantities of these products.

Corn processing can be grouped into several basic operations. These are (1) Milling and component separation, (2) Production of starch and modified starches, (3) Sugar and syrup manufacturing, (4) Oil expelling and refining, and (5) By-product production. The processes all require equipment of special design and are as diversified as the names of the operations imply. Along with this equipment, tonnage quantities of numerous chemicals are required for tailoring the corn kernel into scores of products. In the choice of materials compatible with these process environments, a knowledge of corrosion fundamentals is a necessity.

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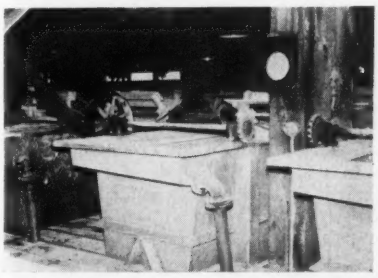

Figure 1—Germ separator cells of welded Type 316 stainless steel in foreground. An outdated cast iron cell is shown at left.

Milling and Component Separation

Milling and component separation involves mechanical processes that split the corn into starch, gluten, germ, and fiber. Warm water at temperatures of 105-120 F with a minimum of 0.1 percent sulfur dioxide by weight is required in the selective prevention of undesirable microorganism formation and in facilitating milling by softening the grain. This severe reducing environment provides the major source of corrosion problems in the initial steps of corn processing.

Since World War II, Type 316 stainless steel has been used extensively in modernizing to replace equipment made mostly from wood and cast iron. Process storage tanks, germ separation cells (Figure 1), and liquid transfer lines are examples where the superior corrosion resistance of Type 316 stainless steel is utilized. The molybdenum content of this alloy furnishes the needed resistance to pitting by sulfur dioxide. On several occasions, Type 304 stainless steel, mistakenly identified as "316," has pitted in less than six months after installation. Figure 2 shows such a failure when a Type 304 stainless steel Van Stone end was welded to a Type 316 stainless steel tube. Numerous pits and perforations soon developed in the flanged section. To guard against using Type 304 stainless steel where "316" is required, the construction and maintenance workers have been instructed in the use of spot testing kits to check qualitatively for molybdenum. By this method, costly failures which can result in lost production and represent high replacements costs are averted.

Another serious problem resulting from sulfur dioxide fumes is the etching and deterioration of concrete structures. Cases can be cited where attack on concrete ceilings and walls has exposed the structural steel (Figure 3). With modernization of the older equipment of open design, this problem is minimized through nearly complete removal of corrosive fumes from the affected areas by an extensive network of fume ducts. During the past few years, stainless steel has been widely used for this purpose. Rigid polyvinyl chloride sheet and polyester laminates are now in limited use and will no doubt partially replace the more expensive stainless steel alloys for vapor ductwork in new construction.

One of the most troublesome problems in handling sulfur dioxide and sulfur trioxide arises during absorption of these gases to form "steep acid." The gases pass through wooden absorption towers arranged in series and connected with Type 316 stainless steel tubing. Accelerated attack from intergranular corrosion caused by condensation of sulfurous and sulfuric acids in the weld zones caused tube failure within six to

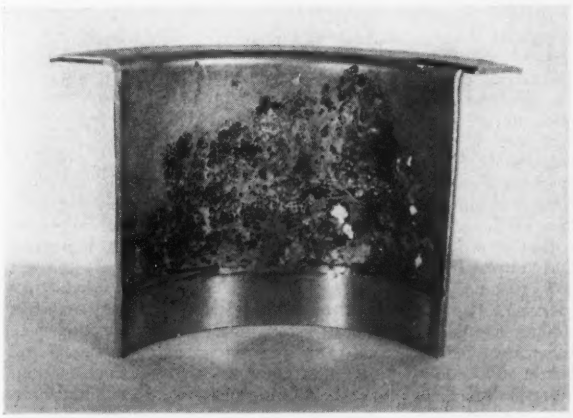

Figure 2—A Type 304 stainless steel stub end corroded to failure in sulfurous acid fumes. The uncorroded section is Type 316 stainless steel.

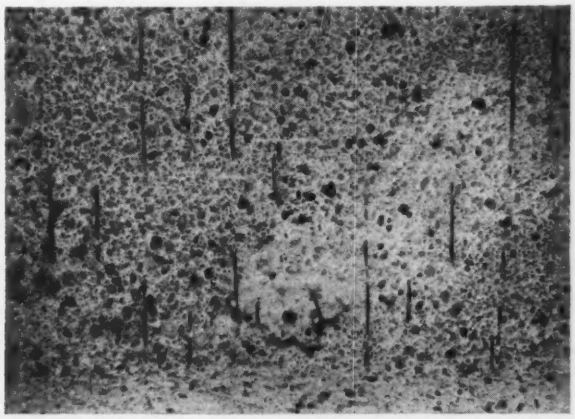

Figure 3—Severe etching of concrete from sulfurous acid fumes resulting in the exposure of reinforcement rods.

RICHARD BENES is corrosion engineer, Technical Division, Corn Products Company, Argo, Illinois. He attended the University of Illinois, receiving a BS in chemical engineering in 1953. At the present he is engaged in corrosion testing and evaluation methods. Mr. Benes is a member of AIChE and NACE.

FRED J. HOLSINGER is a corrosion metallurgist with Research and Development Dept., Corn Products Co., Argo, Illinois. He has a BS in chemical engineering, University of Notre Dame (1941), joining his present employer in 1946 after duty with the navy in World War II. He has been active in corrosion work since 1952.

RUSSELL E. PIERSON is employed by the Technical Division of Corn Products Company, Argo, Illinois as a corrosion engineer. He received a BS in chemical engineering from the State University of Iowa in 1952. His experience before joining Corn Products in 1954 includes a tour of duty in the U. S. Army chemical corps. He is a member of AIChE and NACE.

eight months. The use of No. 20 stainless steel alloy or Type 316 extra low carbon stainless steel added several months' life to these sections; however, this was not considered a satisfactory solution to the problem. A pipe section of molded furane resin has been on test in this environment for more than a year. Based on the success of this test, an entire replacement unit for the stainless steel tubing has been ordered.

Suspension of solids in solutions containing sulfur dioxide produce erosion problems in pump and valve parts. Cast CF-8M and stainless No. 20 alloy are the materials generally used to combat these conditions.

The recovery of starch particles in the one to ten micron range from gluten is done in multistage liquid cyclone units (Figure 4). Aluminum bronze, because of its resistance to sulfurous acid, is used for housings and tube sheets in these units. Each unit contains several hundred separate nylon cyclones, each six inches in length. Individual cyclones of hard rubber and phenolic resin were also tested, but were inferior in resisting the abrasive action of hard starch granules.

Also of importance is the increasing

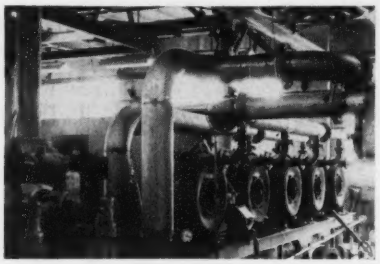

Figure 4—Liquid cyclone units of aluminum bronze. Note extensive use of stainless steel tubing.

interest in using aluminum alloys in the milling process. Results of field tests have been encouraging and it is now evident that aluminum can be safely used in the milder corrosive environments. Several aluminum storage tanks are now in service and a much wider use of this material within a few years is probable.

Starch Production

Once separated from the corn kernel, starch can be modified into countless products. Oxidation, acidification, and other chemical treatments at 110 to 130 F make use of such chemicals as sulfuric acid, hydrochloric acid, and sodium hypochlorite. With the advent of more rigid product specifications and more strict quality control limits, wooden treating tanks are gradually being replaced. For economic reasons, in starch modification processes, it is necessary to use the same treating vessels for various products. A tank in which sodium hypochlorite may be used during one treating period, also has to serve for modifying starch with mineral acids in the production of other products.

Natural rubber, based on its resistance to commercial grade sodium hypochlorite (containing up to 15 percent free chlorine), was originally selected for lining mild steel reaction vessels. After 18 months' service, numerous pinholes and voids developed and the linings were in the advanced stages of failure (Figures 5 and 6). The linings in several tanks were replaced with plasticized polyvinyl chloride sheet. Other than several blisters forming behind this lining, the material has shown no evidence of failure in more than three years. These blisters were caused by the process liquors seeping through pinholes in the seaming between the plastic sheets and liberating carbon dioxide by reacting with the steel tank wall. Trouble spots of this nature have been reduced through the use of rigid acceptance tests for lined vessels. Spark testing and electrical resistance measurements (after total immersion of the lining) are the most used test methods.

A two-ply lining consisting of separate layers of plasticized and unplasticized polyvinyl chloride is now specified for use in modified starch service. The unplasticized portion is in contact with the corrodent and being less permeable than plasticized polyvinyl chloride gives added protection against blister formation. With scheduled inspections and maintenance, this type of lining should have a life in excess of ten years. The use of rigid polyvinyl chloride baffle plates (rather than coated steel baffle sections) and polyvinyl chloride bolts simplifies application, eliminates many sources of possible lining flaws, and reduces initial expenditures.

For dewatering of starch products preparatory to final drying, both horizontal basket automatic batch centrifuges and rotary vacuum string discharge filters are used. As during the milling operations, Type 316 stainless steel is relied upon to resist corrosion in these filtering operations. In the larger diameter centrifuge, however, limiting physical properties of this stainless alloy prevent its usage and a less corrosion-resistant high tensile low alloy steel was selected for the basket and hub assemblies. To obtain the required corrosion resistance, the basket periphery was lined with polyvinyl chloride sheet. The hub was coated with a catalytic cold-set epoxy-phenolic coating. A vinyl protective coating was applied to the filtrate drain holes. A basket of this design (Figure 7) is expected to remain in service from five to ten years.

The rotary filters for special starch products were built initially entirely of Type 316 stainless steel. After four years' operation, pitting of the filtrate tubes and filter bed of one unit was noted. The filter bed subsequently was lined with sheet polyvinyl chloride. Solvent welded rigid polyvinyl chloride pipe was used to replace the filtrate lines (Figure 8). These items have shown no deterioration after two years' service. Bolts and washers of Type 316 stainless steel used on the agitator rocker arm assemblies failed from crevice corrosion in several years (Figure 9). The use of plastic washers or gasketing compounds was recommended to eliminate recurrence of this failure.

Sugar and Syrup Production

In the acid hydrolysis of starches to sugar and syrup, both sulfuric and hydrochloric acids are employed. The reactions take place at 225-275 F and at a pH of 1.2-1.5. After the desired degree of conversion, the acidic sugar solution is neutralized to a pH of 4.5-5.0 with soda ash. For sulfuric acid conversions, equipment fabricated entirely of Type 316 stainless steel has been in service for eight years with only minor corrosion problems. Some intergranular attack in weld zones has occurred. Where this has taken place, replacement is made with Type 316 extra low carbon stainless steel which is more resistant to intergranular corrosion.

In the hydrochloric acid conversion process, the problems involved are more severe and in several instances the high nickel-molybdenum alloys are required to successfully resist corrosion. However, batch converters made of acid resistant bronze have proved entirely satisfactory because a highly protective carbonaceous deposit forms inside the unit. In the more modern equipment used in sugar and syrup production—the continuous converter—the high degree of turbulence encountered sweeps clean the reaction chamber walls and does not allow formation of such a protective deposit. For this

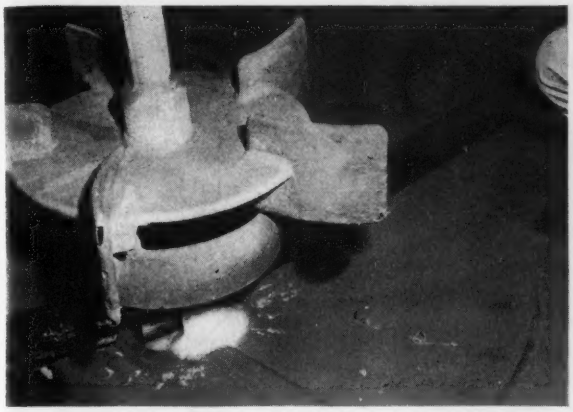

Figure 5—Failure of natural rubber lining on mild steel agitator turbine in starch treated with sodium hypochlorite.

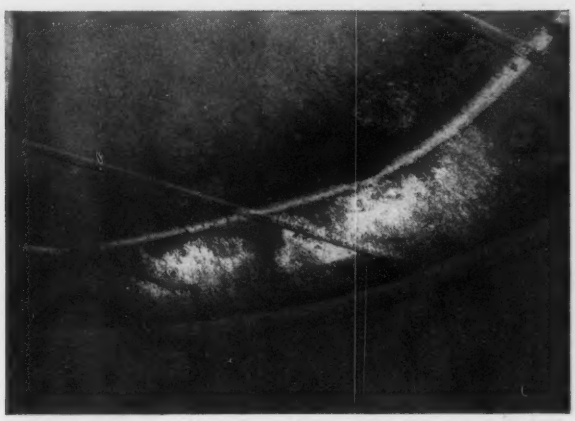

Figure 6—Ceiling of rubber lined starch treating tank. Deterioration of the lining from chlorine vapors is visible.

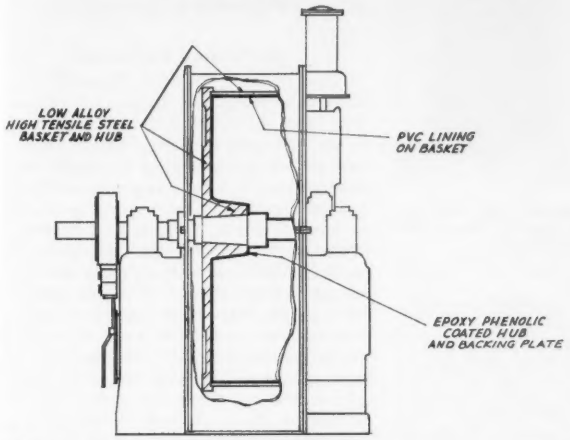

Figure 7—Sections of horizontal basket centrifuge indicating areas where polyvinyl chloride sheet and protective coatings were used to reduce corrosion attack.

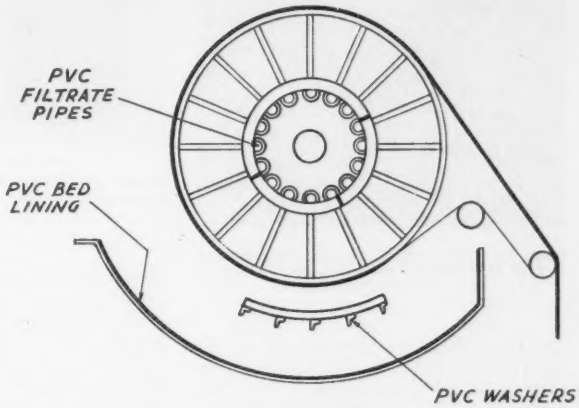

Figure 8—Rotary vacuum filter showing extensive use of polyvinyl chloride as replacement for Type 316 stainless steel.

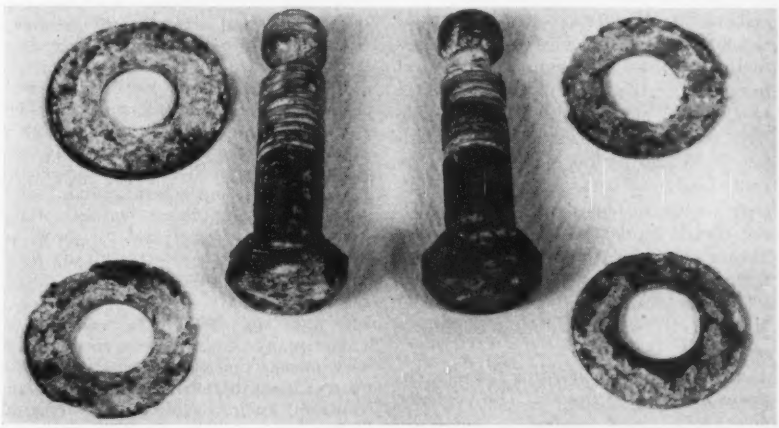

Figure 9—Failure of Type 316 stainless steel bolts from crevice corrosion in a starch slurry containing traces of acid chlorides.

highly corrosive condition, it was necessary to use Hastelloy C[1] which after eight years' service is still in excellent condition. This reaction chamber replaced a silicon bronze unit which failed in less than one year.

[1] Trade name of Haynes Stellite Company, Division of Union Carbide and Carbon Corporation.

In the next process step, neutralized sugar liquors are concentrated in triple effect evaporators. Both Types 304 and 316 stainless steel and copper have been used for evaporator tubes. Service life for stainless steel tubes was originally estimated at 15 to 20 years. Considerably shorter service life has resulted however, since the vapors from the first and second effect evaporators contain a small percentage of acid chlorides which promotes stress cracking. The failure generally occurs in the areas adjacent to the tube sheet, where high stress levels from tube rolling operations are encountered.

A few cases of cracking along the sides of the tubes also have been reported (Figures 10 and 11). Reported failures of these types have taken place in from two to ten years, depending on the degree of conversion and hence the variable concentration of acid chlorides present in the vapors.

Useful life of copper evaporator tubes ranges from five to seven years. Failures result on the vapor side from erosion-corrosion, generally in the tube sheet area (Figure 12). On the product side of the tubes however, no appreciable corrosion occurs because of the formation of a protective film during normal processing operations.

Several inclined tube evaporators (Figure 13) still in service present tube replacement problems unlike those with the conventional vertical tube evaporators. In these units, the total volume of condensate must flow over the few tubes

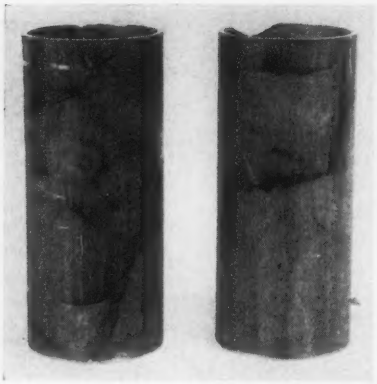

Figure 10—Stress cracking of Type 316 stainless steel tubes in a corn syrup evaporator. Failure of this particular tube resulted within two years.

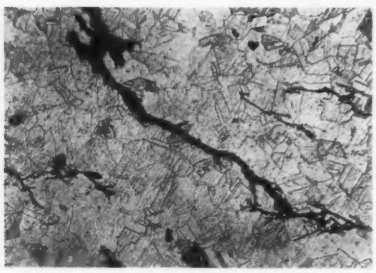

Figure 11—Typical photomicrograph of stress cracks in Type 316 stainless steel tubing. 150X reduced to 50X.

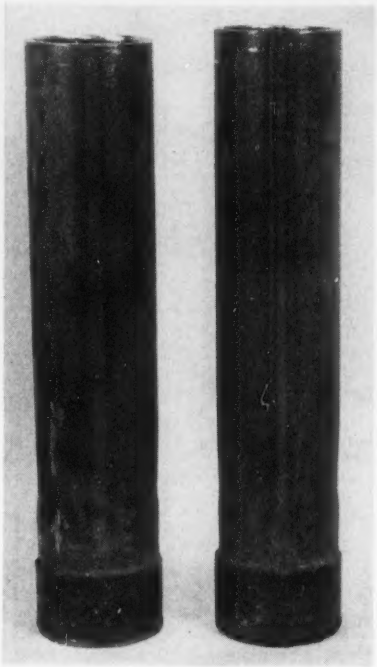

Figure 12—Copper tube removed from corn syrup evaporator after five years' service. Note erosion-corrosion attack.

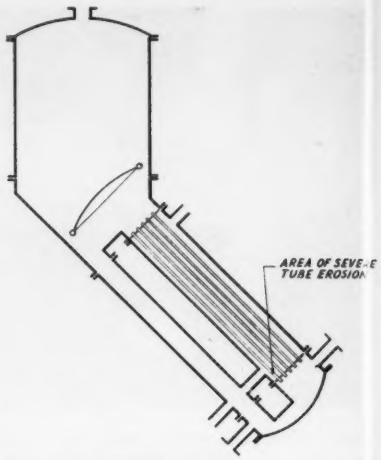

Figure 13—Cross section view of inclined evaporator showing area of severe erosion.

contained in the lowest section of the bottom tube sheet. In that area, the frequency of replacing copper tubes, due to severe erosion, was two to three times that of the other tubes. The problem was resolved by specifying thickened end or dual gauge tubes in the lower one-third of the tube sheet where the greatest erosion occurs. A 60 percent increase in wall thickness in the critical area was obtained for a 15 percent increase in cost.

Although in common usage, neither stainless steel nor copper are considered entirely satisfactory for evaporator tubes for concentrating sugar and syrup liquors obtained from the hydrochloric acid conversion process. Because of stress cracking, service life of stainless steel is unpredictable and its use involves certain risks. The relatively short life of copper means that costs are high both for maintenance and in terms of lost production. Recent field tests with copper-nickel, copper-aluminum, and copper-silicon alloys have indicated that one of these materials may provide a considerable increase in service life and will make possible the eventual replacement of all stainless steel and copper tubes.

Corn Oil Expelling and Refining

The corrosion problems in corn oil processing deal mostly with the expelling and refining equipment. In oil expeller operation, the clarity of the crude oil governs the degree of purification treatment required in later processing. Very close tolerances must, therefore, be maintained between oil expeller bars to prohibit solids from entering the crude oil stream. High carbon steel hardened to 750 Brinell eroded excessively within three to four months and allowed whole germ to pass into the crude oil. Nitriding the steel to form a harder wearing surface did not increase service life enough to warrant the cost of the additional surface hardening.

Thus far, the material that is the most satisfactory for this service is "Haynes Stellite" No. 1[2] applied over mild steel bars. Service life has now been extended to 15 to 18 months with a considerable reduction in maintenance costs. Regrinding the dulled Stellite surface allows repeated usage of bars and results in additional savings. This case illustrates that the use of harder wear surfaces is not always the proper method of attacking abrasion problems. A softer but tougher alloy (hardness of Stellite No. 1 is about 500 Brinell) was required to eliminate frequent repairs.

Bleaching and deodorizing the oil completes the refining process. The tendency of the oil to become turbid or chemically unstable from trace quantities of metallic contamination limits the selection of construction materials for these operations to those which are highly corrosion resistant. For this reason, equipment which contacts the purified corn oil in the final processing is made of Type 316 stainless steel.

[2] Trade name of Haynes Stellite Company, Division of Union Carbide and Carbon Corporation.

By-Product Production

Two of the principal by-products of corn processing are steepwater and animal feeds. Steepwater, the liquor drained from the corn steeping vessels prior to milling, is concentrated in triple effect evaporators before usage in antibiotic production or as a high protein additive to animal feeds. Evaporators with cast iron bodies, copper tubes and tube sheets were standard in the industry for three decades. Replacement or major repair of such equipment was required in approximately five years because of erosion-corrosion failures. The necessity for descaling the evaporator tubes with acid cleaners also contributed to the short life of copper and cast iron components. In contrast, both Types 304 and 316 stainless steel equipment have been in service in steepwater evaporators since 1946 without appreciable wear.

Animal feed combines the gluten, fiber, germ flake, and a portion of the steepwater into a product containing from 21 to 42 percent protein.

Type 316 stainless steel is the most suitable alloy for the moisture expellers, mixer boxes, and wet feed conveyors in contact with the undried materials coming directly from the milling and separation processes and which contain sulfur dioxide. After complete removal of this corrodent, mild steel and cast iron are used successfully in dryers and dry product handling equipment.

Summary

For many of the highly corrosive environments, the standard construction materials of the early chemical industry—wood, cast iron, and copper—are gradually giving way to the more corrosion resistant alloys and metal substitutes for new and replacement equipment in the wet milling industry.

In the past 15 years, the stainless steels, plastics, and specialized organic coatings have contributed greatly in reducing maintenance costs and extending the life of existing equipment. Of equal importance is the improvement in product quality and in manufacturing operations made possible with these materials.

NATIONAL ASSOCIATION of CORROSION ENGINEERS

Application Techniques, Properties And Chemical Resistance Of Polyethylene Coatings

NACE TECHNICAL COMMITTEE REPORT
Publication 59–7

A Report of NACE Task Group T-6A-5 on Polyethylene*

Abstract

The chemical resistance of polyethylene coatings at 75 F and 130 F is reported for 39 acids, 4 alkalies, 12 salts, 33 organic materials and solvents, 6 oxidizing agents, and for tap, distilled and sea water. Physical properties of polyethylene coatings also are given.

The forms in which polyethylene resin is available is considered briefly. Other topics discussed include effect of compounding, application procedures, surface preparation, heating, spraying, test methods, and application hazards. 5.4.5

Foreword

COMMITTEE T-6A was organized for the purpose of assembling and disseminating to the corrosion engineer factual and quantitative data on the performance and limitations of the various organic materials that are successfully used in coatings and linings. While every attempt is made to be factual in the coverage of application and chemical resistance tables and physical properties, it nevertheless should be recognized that changes in compounding formulations of organic materials for coatings or linings enhance or detract from their inherent corrosion resistance characteristics. Under such circumstances, these reports and recommendation lists must be viewed as showing average properties only. The possibility of changes in chemical and physical characteristics as produced by various manufacturers or applicators of the material should be kept in mind.

Definition

Polyethylene (this report refers only to the use of low density polyethylene) is a linear polymer of a partially crystalline structure. The low cost of the resin and excellent chemical stability at ambient temperatures have resulted in many attempts at producing a satisfactory coating with the material. Numerous trials of solution and solvent application of the resin have been made but with little commercial success. Emulsion coatings of the resin have produced obstacles which thus far have proved too difficult to surmount for application of the unmodified resin. Sheet lining with the material has proved difficult due to the lack of an adequate adhesive and the high thermal expansion characteristics of polyethylene.

Like most resins, polyethylene is available in powdered form, and as such, has provided a means of applying a coating of 100 percent solids with a minimum of difficulty. This report presents only data derived from the application of the resin by heating and solidifying in place.

Three methods of applying the polyethylene are in current use in volume. One is by melting the resin and contacting the article to be coated with the molten resin. Another is by heating the object to be coated to a temperature above the melting point of the polyethylene and immersing the article in a fluidized bed of powder. Lastly, a method of flame spraying the polyethylene directly onto a metal surface is available. The fluidized bed principle probably supplies the best of the polyethylene coatings but is limited in the size of the object which may be coated. Flame spraying of polyethylene must be used on larger objects in the field. Only one side of the metal plate may be coated with polyethylene by the flame spraying method.

Summary of Properties

Polyethylene coatings provide no panacea in the coatings field. The coatings are applied in heavy film thicknesses but cannot be assumed to have the impermeability of a calendered film of equal thickness. The excellent resistance of solid polyethylene to all chemicals but strong oxidizing agents, oils, and chlorinated and aromatic solvents has invited the use of polyethylene coatings in many exposures where they should not be employed. On the other hand, the use of polyethylene coatings for general service on production items for the American market has not been fully exploited. Fluidized bed coating of articles on a production basis should provide an extremely durable, economically attractive coating.

The thermal properties of polyethylene are poor and the formulation of a coating material adds little in this respect. Service above 130 F is to be avoided or carefully analyzed before the exposure. Coatings other than polyethylene are available for service above ambient temperature and their use is recommended unless some unique property of the polyethylene is desired.

The electrical insulating properties of the polyethylene coatings applied today are good.

The abrasion resistance of polyethylene films is reported to be poor but coatings applied by the available methods offer long life under conditions of severe wear, possibly as a result of the heavy, resilient film provided. Steel floor plates coated with 60 mils of flame-sprayed polyethylene have been exposed to heavy personnel traffic in an area of chemical spillage for three years without exposure of the substrate.

Patching of a damaged polyethylene coating is readily achieved by sanding or blasting the area to feather out the existing coating and to prepare the substrate for recoating. Care must be exercised in application of the patch coating to prevent burning of the existing film. With care the original coating can be brought to the molten state and fresh resin fused into the film without defects.

Forms Available

Low density polyethylene resin is available in a variety of molecular weights which provide variation primarily in the physical properties of a film produced from the resin. There is some difference in the range of molecular weight and susceptibility to oxidation of resins produced by the various manufacturers, and the proper resin for use as a

* L. S. Van Delinder, Union Carbide Chemicals Company, South Charleston, West Virginia, Chairman.

solid coating material must be indicated by the manufacturers and applicators doing development work on the problem. The resin must be milled to a uniform, small particle size suitable for "fluidizing" or "spraying."

The advent of high density (linear) polyethylene presents a new challenge to applicators of solid polyethylene films. The changes this new material may make in the quality of a polyethylene coating are unknown at this time.

Effect of Compounding

Polyethylene coatings of three general types have been applied. The unmodified polyethylene coatings were found to be too permeable, too highly stressed, and too susceptible to stress-cracking to be fully successful. The addition of carbon black or other inert fillers improved the characteristics of the film, but even greater improvements were realized by the addition of "plasticizers," such as polyisobutylene. Consequently, the improved product available today is a black, heavy film of polyethylene containing a number of stabilizing agents, pigments, adhesion improvers, and a small portion of an elastomeric constituent. A coating consisting of a polyethylene and asphalt mixture has been imported to this country for application by hot spraying, but does not fall under the category of coatings covered by this paper.

Resistances

Physical Properties

A hot applied polyethylene coating is not entirely homogenous, and consequently, a description of the physical properties of an applied film are not precise. The amount of plasticizer, pigment, or other additive in the resin will also affect the ultimate properties of the coating. The molecular weight of the resin used to prepare the coating may be altered at the applicator's discretion and a noticeable change in the physical properties will result.

TABLE 1—Physical Properties of Polyethylene Coatings

Property	Value or Designation	
Thermoplastic (TP) or Thermosetting (TS)	TP	
Specific gravity	0.92–0.94	grms/cc
Tensile strength	2300–3100	lb/sq inch
Elongation	200–600	percent
Hardness	R_{25}–R_{35}	Rockwell
Flexural Strength	1500–1700	lb/sq inch
Refractive index	1.52	n_D^{25}
Thermal expansion	64×10^{-5}	inch/inch/°C
Thermal conductivity	7×10^{-4}	cal/sec/cm/°C/cm
Specific heat	0.55	cal/°C/grm
Resistance to heat (continuous)	80	°C
Softening point	100–115	°C
Flammability	1.1–1.4	inch/min
Water absorption	Nil	24 hr immersion
Moisture vapor permeability	0.1	gr/100 sq inch/day
Dielectric strength	1000–1500	V/mil. (short time)
Dielectric constant	2.3	1–100 Mc/s
Power factor	0.0005	1–100 Mc/s
Volume resistivity	3×10^{17}	ohms/cc
Weathering (pigmented)	Good	
Odor	None	

For the above reasons, the properties of solid, unmodified polyethylene should not be used as a standard for an applied coating. One applicator supplies the values shown in Table 1 as typical of polyethylene flame-sprayed coatings.

One characteristic of polyethylene coatings should not be ignored—the antisticking qualities of the resin. Although Teflon and the silicones present the ultimate in this property, polyethylene has good resistance to the adhesion of foreign materials, and the much lower cost of the polyethylene coating makes the use of this material attractive where practicable.

Another property of value is the excellent low temperature stability of polyethylene. The material retains good ductility and resistance to fracture by impact at temperatures as low as minus 60 F.

Chemical Resistance

The exterior surface of a heavy polyethylene film presents essentially the same chemical resistance as solid polyethylene construction. However, two factors must be recognized as affecting the service life of the coating. Stresses are "built in" a hot applied, polyethylene film, and proper compounding of the product is required to mitigate this problem. Table 2 shows the general resistance of polyethylene coatings to a wide variety of exposures with those exposures marked where stress-cracking of an unmodified film might occur.

Proper compounding alleviates this problem to a great extent as described under the heading "Effect of Compounding." Another method of combating this problem is the quenching of the polyethylene while the film is still hot. This can be accomplished from the back side of a plate and is reported to reduce the stresses present in the cold film.

Secondly, the polyethylene lattice is subject to permeation by molecules of small size. This means that, although the coating is unattacked by certain exposures, the selective absorption of some

TABLE 2—Chemical Resistance of Polyethylene Coatings

CORROSION MEDIA	Temperature 75F	Temperature 130F
Acids:		
Acetic, 10%	R	R
Acetic, glacial	LR+	NR
Acetic anhydride	R	....
Benzene sulfonic	LR	....
Benzoic	R	....
Boric	R	....
Butyric	NR+	....
Carbonic	R	R
Chloracetic	NR	....
Chromic, 10%	R	R
Chromic, 50%	R	R
Citric	R	....
Fatty acids (>C_8)	LR	....
Fluosilicic	R	....
Formic, 100%	R	LR
Formic to 50%	R	LR
Hexoics	NR	....
Hydrobromic, 50%	LR	LR
Hydrochloric, 36%	LR*	LR*
Hydrochloric, 10%	LR*	LR*
Hydrofluoric, 75%	LR	LR
Hydrofluoric, 40%	LR	LR
Hypochlorous	NR	....
Lactic	R	R
Maleic	R	....
Nitric, 95%	NR	NR
Nitric, 40%	LR	LR
Nitric, 20%	LR	LR
Nitric, 5%	R	R
Octanoics	NR	....
Oxalic	R	....
Phosphoric, 90%	R	NR
Phosphoric, 30%	R	R
Picric	LR	NR
Sulfuric, 98%	NR*	NR*
Sulfuric, 70%	LR*	NR*
Sulfuric, 10%	R	R
Sulfurous	R*	LR*
Tannic	R	R
Alkalies:		
Ammonium hydroxide	R*	....
Calcium hydroxide	R	RR*
Potassium hydroxide	R	R
Sodium hydroxide	R	R
Salts:		
Aluminum chloride, sulfate	R	R
Ammonium chloride, sulfate	R*	R*
Barium chloride, sulfate	R	R
Calcium chloride, sulfate	R	R
Cupric chloride, sulfate	R	R
Ferric chloride, sulfate	R	R
Magnesium chloride, sulfate	R	R
Nickel chloride, sulfate	R	R
Salts (Continued):		
Potassium chloride, sulfate	R	R
Silver nitrate	R	....
Sodium chloride, sulfate	R	R
Stannic chloride	R	R
Organic Materials and Solvents:		
Acetone	NR	NR
Aldehydes	LR+	....
Amines	LR*	....
Aniline	LR	NR
Animal oils	NR	NR
Beer	R	....
Benzene	NR	NR
Butyraldehyde	LR+	....
Carbon tetrachloride	NR	NR
Chelating agents	NR	....
Chloroform	NR	NR
Cider	R	....
Cyclohexanone	NR	NR
Detergents	R+	....
Dibutyl phthalate	R	....
Diethylene glycol	R	LR
Esters (C_4)	R	....
Ethylene dichloride	NR	NR
Ethylene glycol	LR	....
Formaldehyde	LR	....
Glycerin, 50%	R	R
Glycols (C_4)	R	R
Methanol	LR	....
Methyl bromide	R	....
Mineral oil	NR	NR
Phenol	LR*	NR
Tanning extracts	R	R
Trichlorethylene	NR	NR
Vegetable oils	NR	NR
Vinegar	R	R
Whiskey	NR	....
Wine	NR	....
Xylene	NR	NR
Oxidizing Agents:		
Chlorine	NR	NR
Chromic acid	(see acids)	....
Hydrogen peroxide	R	LR
Iodine	NR	NR
Calcium hypochlorite	NR	....
Sodium hypochlorite	NR	....
Water:		
Tap	R	R
Distilled	R	R
Sea	R	R

CODE: R–Recommended; LR–Limited recommendation; NR–Not Recommended.
* Permeation of the film by one constituent may occur.
+ Stress-cracking of the film may occur unless properly modified.

compounds will take place. Thus, dry HCl and dry SO_3 will eventually reach the substrate from aqueous solutions of these gases. Ammonia will be absorbed from ammonium hydroxide solutions similarly. This does not mean that the polyethylene coating would not have a justifiable economic life in such an exposure, but it does point out the need for a proper evaluation of the coating and it characteristics before recommending its use in a given exposure. An asterisk is used to mark those exposures listed in Table 2 where absorption of a dry gas by the film is known to occur.

There are a number of factors to be considered when selecting a coating for a particular service and Table 2 is intended only to suggest possible uses for the coating. Tests should be conducted by exposure of a test panel of the coating in the environment for a minimum of sixty days before any recommendation is made. (See "Test Methods" for the evaluation of a prepared coating.)

Application

Application of the polyethylene resin to a hot surface or by a flame spray method causes an oxidation of the resin at the metal coating interface. This provides a polarity in the molecule not present originally and accounts for adhesive properties of the coating not found in the basic resin. Successive layers of the resin become farther removed from the heat of the metal and form a heavy layer of unchanged resin as a top coating. The oxidation occurring in that resin adjacent to the metal surface is difficult to control properly and constitutes one of the arts in the application of this coating. The coating near the metal surface becomes porous with fine gas bubbles, and careful control of the conditions of application must be maintained to prevent excessive porosity in the coating at this level.

The proper application of this coating demands the proper equipment and a development program with personnel to attain skill in the application procedures. This type of coating application has no relationship to spray coating, welding, or other conventional techniques.

Coating with solid polyethylene is an art. Unless a thorough investigation of the problem coupled with patience in personnel training can be fulfilled, the application of such a coating must be contracted with a reputable applicator experienced in the work.

Surface Preparation

Restrictions on the design and construction of items to be coated with polyethylene are the same as those which apply to the application of other coatings. A minimum of angles and recesses must be built into the design and sufficient radius of curvature provided to allow the proper application of a coating. All areas of the metal should be as smooth as practical. The coating should not be applied over rivited construction.

Surface preparation for the application of the coating by any method requires that the metal be free of all oil, scale and extraneous matter. As in the case of almost any coating application, a sandblasted surface is desirable from the standpoint of cleaniness and profile pattern of the surface. The coating may be applied to properly pickled and cleaned surfaces, but non-polar character of this resin does not provide a high degree of adhesiveness to a smooth steel surface. The adhesion obtained is not the equivalent of that obtained on a sandblasted surface. A "white metal" blast corresponding to NACE No. 1 pattern* should be obtained using the appropriate size of sand, slag or grit. This provides an adequate surface profile to "anchor" the resin to the surface and increase the apparent adhesion. As in the case of sandblasting for the application of other coatings, the area prepared should be no larger than that which can be coated before any oxidation of the surface occurs.

It has been found for reasons not fully understood, that the application of a polyvinyl butyral wash primer containing chromate salts to a steel surface preceding the coating operation increases the adhesion of the coating in many instances. Where the surface is being coated by flame spraying, the high temperatures produced by direct impingement of the flame must partially decompose this organic metal-pretreatment, but improved adhesion is reported by a majority of applicators. Other primers have been found to be of questionable value beneath a polyethylene coating. The use of the wash primer immediately following the sandblasting operation also allows an extended time interval before overcoating with the final film.

Application of the coating on sandblasted aluminum and stainless steel surfaces has been made with good results. If it is desirable to coat a copper alloy surface, the metal must first be treated with one of the proprietary oxidizing salts available to produce a tightly adherent oxide coating on the surface. The polyethylene will then adhere to the surface.

Heating

After preparation of the surface, the metal must be preheated before application of the resin. In the flame spraying procedure, the application torch is adjusted to provide a flame suitable for rapid heating of the metal. An area of 10-15 square feet of the surface is preheated to approximately 425 F by moving the flame steadily back and forth across the piece. When a temperature-indicating crayon mark on the surface shows the appropriate color, the section is ready for an application of the resin.

When the dip coating process is employed, the object is preheated to essentially the same temperature in a controlled temperature oven, then removed to the coating chamber. Flow coating or slush molding on the interior of some objects is accomplished by heating from the exterior while rotating the object containing resin particles.

*NACE Publication 50-5, Recommended Practices for Surface Preparation of Steel, 1950.

Spraying

Polyethylene application by the exposure of articles in a fluidized bed requires specialized equipment best used by the commercial coating applicator. A better quality of coating is probably achieved by this method, but the size of the article establishes a definite limit to the method.

For field applications or shop applications on objects of larger size, the flame spray method must be employed. This requires an air supply, heating gas supply, a powder dispenser, and an applicator's gun. The last two items are specialized pieces of equipment designed for this work and makeshift items cannot be substituted for this equipment. The gun has controls for varying the flame, the volume of air, and the quantity of powder ejected from the nozzle. The surface of the object is first preheated as described under the heading "Heating." The flame of the gun is then adjusted as to intensity and distance from the work. The dry powder is then forced through the nozzle from the dispenser. A first pass over the metal provides a bonding coat of less than 10 mils. Subsequent passes are made to build the film to thicknesses of 30-80 mils. The edges of the working area previously preheated are feathered out and built-up during coating of the adjoining area of work.

The completed coating should have a black, semi-glossy, dense appearance on the surface. The coating thickness should never exceed 120 mils.

Test Methods

The thickness at which polyethylene films are applied and the ductility of the finished coating make the inspection of these coatings different from that of conventional films.

As a preliminary test panel or as a control panel during application on the job, select a section of ⅛-inch or heavier metal approximately one foot square. After appropriate surface preparation, mask off a portion of the plate with a silicone grease or clay slurry. Have the polyethylene coating applied over all of the panel in the conventional manner.

The coating on the test panel and on the completed work should have a smooth, semi-glossy, uniform appearance with a minimum of sags and overspray, and with no evidence of burning of the resin. Thickness measurements of the coating should indicate the specified thickness ± 10 mils. A properly calibrated magnetic gauge is acceptable for making this measurement.

Pinhole testing should be conducted employing a static spark tester of 30,000 to 60,000 volts. Porosity testing by filling the vessel or immersing the object in water containing a wetting agent and measuring current flow may be acceptable under certain circumstances. The heavy thickness of the film precludes the use of wet continuity testers.

On the test plate, make two parallel cuts ¼ to ½ inch wide completely to the substrate with a knife beginning well

over on the masked area. Peel this strip back starting in the masked area, and when the untreated base metal is reached, note the adhesion of the film to the metal. The adhesion of the film to the substrate should exceed the cohesive strength of the resin particles and small tufts of resin should be left on the surface of the metal. Make two parallel cuts in the area of the coating applied on clean metal. It should be impossible to peel a strip from this area. The resin should break apart in small chunks without stripping from the metal.

Taking the strip of film removed from the test plate, bend double and crease with fingers. No cracking of the film should occur in this 180-degree bend. Similarly, pulling apart a strip of the film should cause a reduction in cross-section at the point of rupture indicative of good ductility.

The strip removed from the test panel should be examined in cross-section using a 5X to 20X glass. The 10 mils of coating adjacent to the substrate may present a porous appearance but the remainder of the film should be dense and uniform with no air pockets or unfused resin evident.

If flame spraying of the resin is being employed in the field, a more severe test of the operator's skill may be devised by welding a sheet metal L on a base metal plate and having the interior surface coated. The overlap of the coating near the junctures of these plates should be examined carefully for flow-in and adhesion between the layers of applied resin.

Hazards

Although experience has revealed no special hazards attending the application of polyethylene, the normal precautions for coating work should be observed. A small amount of decomposition products may be released from the hot resin which should not be inhaled by the operator in a confined space. The use of air masks is recommended.

Prolonged exposure to the bright flame of the operator's gun may cause eye strain and tinted goggles are a recommended safeguard.

Obviously, the field application of this coating should not be permitted in hazardous areas and the vessel or other enclosed space should be tested before lighting the gun.

NATIONAL ASSOCIATION of CORROSION ENGINEERS

Amount of Annual Purchases Of Corrosion Resistant Materials By Various Industries

NACE TECHNICAL COMMITTEE REPORT
Publication 59–8

A Report of NACE Technical Unit Committee T-3C on Annual Losses Due to Corrosion*

Abstract

NACE Technical Unit Committee T-3C mailed out 1900 questionnaires to determine the annual purchase of corrosion resistant materials by various industries. Results given in the 205 questionnaires returned are tabulated and extrapolated. Cost data for corrosion resistant materials are reported for the following basic industrial groups: (1) petroleum refining, (2) chemicals, (3) pulp, paper and paperboard, (4) electric light and power, (5) crude and refined petroleum products, (6) crude petroleum and natural gas production, and (7) gas utilities. Annual material purchases made by each group are indicated for each of the following corrosion resistant materials: coatings, mastics, tape, rubber linings, plastic linings, other linings, rigid or reinforced plastics, special corrosion resistant metals, and wood, glass or ceramic materials. 1.2.2

Introduction

NACE COMMITTEE T-3C mailed out 1900 questionnaires to determine the annual purchases of corrosion resistant materials by various industries. Mailings of the questionnaire were made to maintenance engineers of various companies throughout the country, to NACE members, and to maintenance engineers of companies and industries not covered by the first two mailings.

Questionnaire

Only actual material costs were considered on the questionnaire so as to limit the variables inherently connected with a study of this type. The material classification from the Materials of Construction Report as published in Chemical Engineering, December, 1952 was used as a guide and modified. The B. F. Goodrich Industrial Products Technical Department and the Battelle Memorial Institute helped to set up standardized definitions to make up the following list:

1. Coating materials: applied as liquids by brush, spray or dip (e.g., maintenance paints or plastisols, vinyls, epoxies, phenolics, etc., in solution form).

2. Mastics: heavy bituminous base liquid or paste compositions applied by heavy brush, trowel, or air gun.

3. Tape: used for protective service against corrosion.

4. Organic lining materials: rubber and rubber-like materials (which are vulcanizable) and plastic linings.

5. Other lining materials: wood, glass, ceramic, and metal, etc.

6. Materials used as a substitute for steel pipe, ducts, or tanks: plastics, corrosion-resistant metals and metal alloys, and other materials such as equipment made entirely of wood, glass, ceramics, etc.

A total of 205 questionnaires of 1900 mailed were returned. One hundred questionnaires were not usable in this survey because they lacked purchase data. Although the percentage of the returned questionnaires was small, on an employment basis (number of production workers in each plant was requested), the questionnaire returns cover some 4 to 20 percent of the national employment for the selected industries. Industry totals given in the report are extrapolated from the data given in the return questionnaires.

One important factor in the low response percentage was that many companies do not keep records of the cost for corrosion resistant materials. Also, many questionnaires undoubtedly went to persons who did not have access to company figures on corrosion control costs.

* S. K. Coburn, Association of American Railroads, Chicago, Illinois, Chairman.

TABLE 1—Annual Purchases of Corrosion Resistant Materials by Industries

ITEM	AMOUNT OF ANNUAL MATERIAL PURCHASED BY VARIOUS INDUSTRIES						
	Petroleum Refining	Chemicals	Pulp, Paper and Paperboard	Electric Light and Power	Crude & Refined Petroleum Products	Crude Petroleum & Natural Gas Production	Gas Utilities
Coatings	$19,000,000	$15,000,000	$3,600,000	$1,500,000	$620,000	$8,935,000	$11,000,000
Mastics	550,000	1,300,000	670,000	325,000	575,000	1,300,000	5,200,000
Tape	95,000	350,000	15,000	75,000	195,000	245,000	1,400,000
Rubber Linings	50,000	4,800,000	1,250,000	130,000	**	*	25,000
Plastic Linings	25,000	1,000,000	660,000	700,000	**	175,000	*
Other Linings	25,000,000	2,725,000	575,000	165,000	**	*	*
Rigid or Reinforced Plastics	100,000	2,900,000	310,000	100,000	12,000	980,000	400,000
Special Corrosion Resistant Metals	10,000,000	12,300,000	5,000,000	3,200,000	12,000	700,000	30,000
Wood, Glass and Ceramic Materials	*	250,000	1,800,000	2,000,000	**	120,000	*

* Under $15,000.
** Under $10,000.

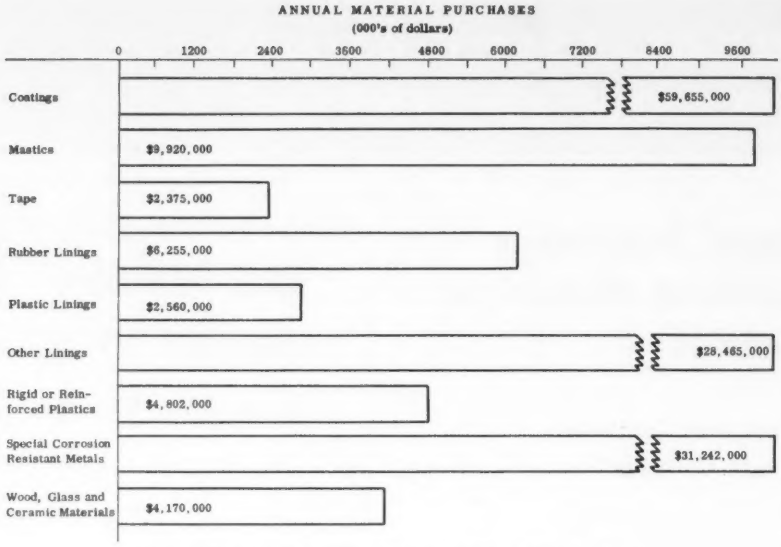

Figure 1—Total annual material purchases for seven industrial groups.

Purchases by Industrial Groups

Seven groups of industries were selected. The amount each industrial group spends annually on corrosion control materials was tabulated for the following material classes: coatings, mastics, tape, rubber linings, plastic linings, other linings, rigid or reinforced plastics, special corrosion resistant metals, and wood, glass and ceramic materials. This information is given in Table 1.

Group One (Petroleum Refining): includes petroleum refining and the processing of coke and other petroleum and coal products. Questionnaires returned from this group represents 21.3 percent of the total employment force of this group in the United States. The largest expenditures were on linings other than rubber and plastic linings.

Group Two (Chemicals): includes those industries which are primarily concerned with the production of industrial inorganic chemicals, industrial organic chemicals, and soap, cleaning and polishing preparations. The returned questionnaires represented 10.27 percent of the total employment of this industry. The largest material purchases of this group were for coatings and special corrosion resistant metals.

Group Three (Pulp, Paper, and Paperboard): includes pulp, paper, and paperboard mills. Questionnaire returns represented 5.85 percent of industry employment. Table 1 indicates that most purchases were for special corrosion resistant metals.

Group Four (Electric Light and Power Companies): includes only those industries which produce electric power. About 4 percent of the national employment in this industry was covered by the questionnaires returned. This group spent over 3 billion dollars for special corrosion resistant metals.

Group Five (Crude and Refined Petroleum Pipelines): includes companies primarily engaged in transportation of crude and refined petroleum products other than natural gas by pipelines. Representing 21.65 percent of the industry's total employment force, the data reported reveal that material purchases for coatings and mastics were almost equal for this industry group.

Group Six (Crude Petroleum and Natural Gas Production): includes production by oil companies and also contract services. Almost 6 percent of the production workers in this group are represented. The greatest expenditures were for coatings.

Group Seven (Gas Utilities): includes natural gas transportation and distribution. Also included in this classification is mixed manufactured or LP gas production and/or distribution. Returns accounting for 7.33 percent of the industry show that this group spent 11 billion dollars for coatings.

Summary

The total annual purchases of the seven industrial groups have been combined and shown in Figure 1. The largest expenditures were for coatings—almost twice the amount spent for special corrosion resistant metals. The smallest total purchases were for tapes and plastic linings.

NATIONAL ASSOCIATION of CORROSION ENGINEERS

Cathodic Protection Of Process Equipment

A Report Prepared by NACE Task Group T-3G-3*
On Cathodic Protection of Process Equipment

NACE TECHNICAL COMMITTEE REPORT
Publication 59–9

Abstract

Ten case histories are cited to show ways in which cathodic protection principles can be applied to chemical process equipment. Materials and items of equipment for which case histories are reported include a Type 316 stainless steel cooling coil in sodium hypochlorite, lead pipe coils in barium chloride, mild steel in chemically pure 73 percent caustic, reactor cells in 10 percent hydrochloric acid, Type 302 stainless steel in dilute sulfuric acid-organic acid mixture, copper steam coils in zinc chloride, and copper heating coils in ammonium chloride.

Criteria for protection were found to differ considerably from that generally accepted for iron or steel in substantially neutral environments. Although process applications usually require laboratory development, cathodic protection may often provide an economical solution to corrosion problems. 5.2.4

Introduction

CATHODIC PROTECTION of buried or submerged structures has received widespread attention in the last decade. The more conventional applications involve soil (ground waters), fresh or brackish waters, or sea water as the corrosive environment, usually at ambient temperatures. Metals commonly placed under protection in these media are iron and steel, copper alloys and lead.

In the past, slight emphasis has been placed on the utility of cathodic protection in chemical process equipment. This type of equipment usually is fabricated from materials which possess good inherent corrosion resistance (e.g., nickel, stainless steels, copper alloys and high nickel and chromium alloys.) However, all metals are more or less susceptible to some form of localized attack under the proper conditions; pitting, intergranular attack, stress corrosion cracking, dezincification of brass, crevice corrosion, impingement, or galvanic attack are examples of attack that may occur. Since these phenomena all proceed via some electrochemical mechanism, cathodic protection may be of decided benefit if the design of the particular equipment does not restrict current distribution. Product contamination resulting from what might normally be considered insignificant metal dissolution could conceivably be controlled by cathodic protection.

The corrosive media encountered in chemical process streams undoubtedly will include concentrated and dilute acid solutions, probably in combination with organic or inorganic salts, and weak to strong alkalies. Furthermore, chemical processes normally operate at elevated temperatures.

Obviously, cathodic protection is not a cure-all for all the corrosion evils of the chemical industry. However, in numerous cases it has proven to be an effective corrosion control weapon. Most applications in this field of cathodic protection are highly specialized and require substantial laboratory study to clarify polarization characteristics, current requirements, potential-time and ampere-hour effects, and to evaluate galvanic and/or impressed current anode materials. Application of cathodic current to passive metals could instigate cathodic corrosion due to a breakdown of inherent passivity under locally reducing conditions. This factor must be recognized. Where cathodic corrosion is encountered, anodic polarization may prove beneficial.

The purpose of this report is to present several case histories compiled by Task Group T-3G-3 on successful applications of cathodic protection to chemical processing equipment. A survey of the current literature reveals little, if any, information on this subject. The specific corrosion problems which initiated these installations and subsequent cathodic protection design and application factors are not discussed in detail. The general scope of the report is limited primarily to performance data. However, where cost analyses have been made, the appropriate cost reductions attained through cathodic protection are included. The ultimate aim of Task Group T-3G-3 is to stimulate additional research in this particular phase of cathodic protection and, furthermore, to create a greater awareness as to the future possibilities of this technique for corrosion control of process equipment.

Case Histories

1. A Type 316 stainless steel cooling coil in a sodium hypochlorite solution, 11 percent available chlorine, failed in 2 to 3 months. The coil was fabricated from 2½ inch diameter tubing, 9 turns on a 2 foot radius. An impressed current cathodic protection system was installed to alleviate the problem. Four graphite anodes were set 90 degrees from each other inside the coil. A current density of 8 ma/sq ft of cathode surface extended the service life of the cooling coil to more than 5 years.

2. Lead pipe coils in barium chloride treatment tanks were experiencing severe general corrosion. The process conditions included 54 degree Bé zinc chloride plus barium chloride at 200 F. Zinc anodes were installed for cathodic protection. The coil life was doubled (6 months to 1 year).

3. Some experimental studies were reported on protection of mild steel in chemically pure 73 percent caustic plus 0.3 percent sodium chloride at 100 C. Since the tests were of short duration, it is felt that the results reported are merely indicative and supply no absolute basis for specific design recommendations. The corrosion rate of unprotected specimens was 190 mdd while the potential was observed to be —1.26 volts measured against a saturated calomel electrode. Application of protective current at increasing intensity levels revealed a progressive decrease in corrosion rate.

Ma/sq ft	mdd
139	130
245	80
544	25
1087	10

Over this current density range, maximum polarization was less than 50 millivolts. Experiments with steel coupons under tension over the current density range of 0 to 300 ma/sq ft showed no outward or visual evidence of hydrogen embrittlement. Monel anodes used in these tests exhibited non-sacrificial properties when anode current densities up to 3370 ma/sq ft were applied.

4. Severe corrosion of copper and Hastelloy components occurred in 14 reactor cells handling 10 percent hydro-

* A. A. Brouwer, The Dow Chemical Company, Midland, Michigan, Chairman.

chloric acid at 60 C. Heaters failed rapidly and considerable production was lost. Twenty lead disc anodes 16 inches in diameter each weighing 200 pounds were distributed throughout the system (no data given but presumably energized by rectifiers). The initial installation cost was $5000 and operating costs are $1000 per year. An annual reduction of $50,000 in maintenance expenditures was reported plus a 2 percent increase in production.

5. Type 302 stainless steel vessels were used to process a dilute sulfuric acid-organic acid mixture (pH 1.5) at a temperature of 100 C. The units failed in less than 12 months due to intergranular corrosion adjacent to welds. After repairs, an impressed current cathodic protection system was installed in one tank. Annealed and sensitized Type 302 corrosion specimens were placed in and electrically connected to the vessel and a duplicate set was installed in an unprotected tank. A current density of 12 to 15 ma/sq ft was applied to the stainless steel surfaces for a period of 10 months. The sensitized coupons showed good resistance to intergranular corrosion under the applied cathodic current. Weld zone areas in the tank likewise exhibited no significant attack. Furthermore, cathodic corrosion of the gross metal surface was not observed. In the unprotected control tank, sensitized specimens 120 mils thick completely disintegrated in five months. Annealed samples were completely resistant in both tanks. The impressed current Duriron anodes utilized in the installation showed negligible weight loss at an anode current density of 2 amps/sq ft.

It may be of interest to note that anodic polarization in the same current density range (10 to 15 ma/sq ft) also effectively mitigated intergranular corrosion in this particular system.

6. Excessive corrosion of copper heating coils was encountered in zinc chloride concentrating tanks. The process liquor contained zinc chloride, 54 degrees Bé to 74 degrees Bé, and sodium chlorate at a temperature of 104 C to 111 C. Steam circulated through the coils. Zinc anodes were installed for cathodic protection. Although direct savings were reported as indeterminate, the service life of the coils was extended considerably.

7. Several installations of cathodic protection in steel process equipment were reported where the corrosive media was more or less conventional (e.g., brackish water, highly chlorinated water, or concentrated brines.) One aggravating problem involved pitting of a synthesis gas interstage drip cooler handling brackish water. Magnesium anodes were installed at an initial cost of $1250. An annual saving of $2000 resulted. Several raw brine inventory tanks have been receiving cathodic protection from magnesium anode and impressed current systems for nearly 10 years. Current requirements are 10 to 15 ma/sq ft for bare steel surfaces. Savings were estimated at several thousand dollars per year.

8. Corrosion of copper steam coils in zinc chloride and ammonium chloride concentrating tanks caused product contamination (metallic copper) and coloration (blue) problems. Zinc anodes were installed in the zinc chloride tanks and an impressed current system utilizing lead anodes was used in the ammonium chloride tanks. Corrosion control was achieved, product purity improved and an annual maintenance saving of $1200 effected.

9. Copper heating coils failed rapidly in an ammonium chloride boil-down tank. Zinc slab anodes were installed for protection. The service life of the coils was extended several-fold.

10. Nickel lined tanks were used in the production of a high purity crystalline salt. The process liquor was moderately alkaline and ranged in temperature up to 100 C. Although corrosion as normally considered was negligible, superficial localized attack contaminated the crystals with nickel corrosion products. Removal of the impurity was costly, although necessary. A magnesium anode cathodic protection system of somewhat special design was installed in each vessel. Complete control of corrosion and product contamination was afforded. Annual savings through improved operations and increased production exceed $15,000.

Summary

The case histories compiled by Task Group T-3G-3 illustrate that cathodic protection principles can be applied to chemical process equipment. Criteria for protection differs considerably from that generally accepted for iron or steel in substantially neutral environments. Although process applications are quite specific and usually require laboratory development, cathodic protection may often provide an economical solution for aggravating corrosion problems.

NATIONAL ASSOCIATION of CORROSION ENGINEERS

Iso-Corrosion Rate Curves For High Temperature Hydrogen-Hydrogen Sulfide*

NACE TECHNICAL COMMITTEE REPORT

Publication 59—10

A Contribution to the Work of NACE Technical Group Committee T-8,
By E. B. Backensto* and J. W. Sjoberg*

Abstract

Iso-corrosion rate curves were drawn for the 0-5 percent chromium steels and for chromium-nickel austenitic steels in high temperature hydrogen-hydrogen sulfide. These curves show the maximum corrosion rates expected over wide ranges of hydrogen sulfide concentrations and temperatures such as would be encountered in petroleum catalytic refining processes utilizing hydrogen. The pressure variable was confined to the range 175-500 pounds per square inch gauge. 3.4.8

Introduction

HIGH-TEMPERATURE hydrogen sulfide corrosion problems have increased in the petroleum industry with the rapid growth of catalytic reforming. This type of corrosion has been the subject of a number of technical papers during the past three years. An excellent summary of high-temperature catalytic reformer corrosion data was made by Sorell.[1]

The first paper[2] on high-temperature hydrogen sulfide corrosion aimed specifically at catalytic reforming operations was presented to the industry by Socony Mobil Oil Company in October, 1955. Five months later,[3] available laboratory corrosion rate data on carbon and low chromium steels were summarized in a useful plot of iso-corrosion rate curves. Iso-corrosion rate curves for the chromium-nickel austenitic stainless steels were presented two months later.[4] Since that time a number of additional laboratory tests have been made which have yielded further corrosion rate data. This report presents revised iso-corrosion rate curves which cover a greater range of hydrogen sulfide concentration than did the original curves.

New Iso-Corrosion Curves

Figure 1 shows the new curves for the 0-5 percent chromium steels and Figure 2 shows the new curves for the chromium-nickel austenitic steels. The major variables plotted are temperature and hydrogen sulfide concentration with curves showing constant corrosion rate. The pressure variable is confined to the range of 175-500 pounds per square inch gauge because limited data[2] at 0.1 percent hydrogen sulfide by volume and 985 F indicate that corrosion rate is unaffected by pressure in this range. Actually, most of the data below 1000 F were obtained at 450-500 pounds per square inch while above 1000 F they were at 175-200 pounds per square inch. The laboratory technique and test specimens used were described in an earlier paper.[2]

The iso-corrosion rate curves in Figures 1 and 2 represent an interpretation of laboratory tests of greater than 150 hours exposure. These curves show the *maximum* corrosion rate to be expected at a given operating temperature and hydrogen sulfide concentration in a hydrogen-containing stream. Numerous Socony Mobil tests at various locations in commercial reforming units have demonstrated the validity of these laboratory design curves. As discussed in an earlier paper,[5] these curves may not predict the corrosion rate at the outlet of a naphtha pretreater or catalytic desulfurizer because the presence of minor constituents in the stream appears to hinder the corrosion mechanism.

For the 0-5 percent chromium steels (Figure 1), the new curves indicate that, in general, corrosion rates below approximately 0.07 ipy will be obtained at milder conditions than would have been predicted from the old curves. Rates above approximately 0.07 ipy will be obtained at more severe conditions than would have been predicted from the old curves. As before, these curves reach a minimum concentration point and reverse direction. On the old curves these minimum points all fell at about 1000 F. On the new curves these points occur at progressively higher temperature with increasing corrosion rates.

At most conditions the new curves for chromium-nickel austenitic steels in Figure 2 predict somewhat higher corrosion rates than the old curves. These revised iso-corrosion rate curves show a greater tendency than the old ones to reach a minimum concentration point in a manner similar to the 0-5 percent chromium steel curves.

Iso-corrosion rate curves were not prepared for the 7-16 percent chromium steels. Not all the steels represented by this group follow the same pattern over the range of conditions being considered. At many conditions, particularly at high hydrogen sulfide concentrations, these steels show no better corrosion resistance than the lower chromium steels. At mild conditions of temperature and hydrogen sulfide concentration, these steels show increasing resistance with chromium content. For most conditions, the iso-corrosion rate curves in Figure 1 may be used for the group of 0-9 percent chromium steels.

Comparison With Other Investigations

Other investigators[6,7,8,9,10] also have plotted corrosion rate curves. While these curves do not agree exactly, all of them probably would lead to the same conclusion for the selection of materials. The present graphs cover as wide (or wider) a range of temperature and hydrogen sulfide concentration as data reported by others. These charts predict slightly higher corrosion rates than the data of Hur, Deichler, and Worrell.[6] Except at 1000 F where the data are in good agreement, higher corrosion rates are obtained with these charts than the curves published by Dravnieks and Samans.[7,8] These charts, however, predict lower corrosion rates than the data of Bruns[9] or Phillips.[10]

In summary, additional laboratory data have permitted revision and extension of previously published iso-corrosion rate curves for high temperature, hydrogen-hydrogen sulfide. Two new charts are presented: one for the 0-5 percent chro-

★ A paper presented at a meeting of NACE Technical Group Committee T-8, New Orleans, La., October 21, 1958.

* Socony Mobil Oil Co., Inc., Research and Development Laboratory, Paulsboro, New Jersey.

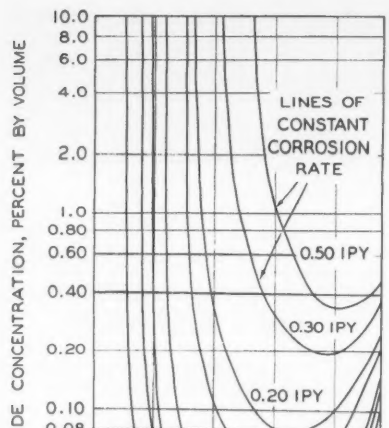

Figure 1—Effect of temperature and hydrogen sulfide concentration on corrosion rate of 0—5 percent chromium steels. Graph is from laboratory data at 175 to 500 psig hydrogen pressure.

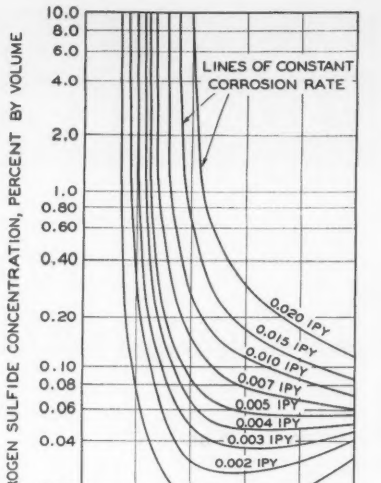

Figure 2—Effect of temperature and hydrogen sulfide concentration on corrosion rate of chromium-nickel austenitic steels. Graph is from laboratory data at 175 to 500 psig hydrogen pressure.

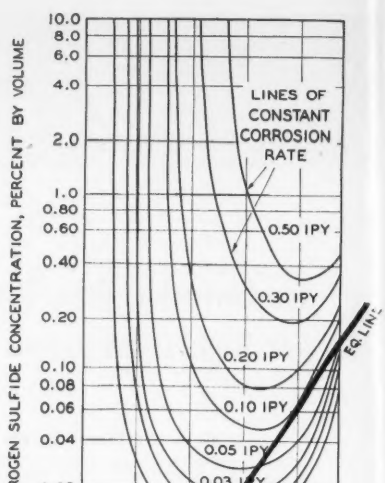

Figure 3—Approximate position of equilibrium line with respect to iso-corrosion lines.

mium steels and another for the chromium-nickel austenitic steels. These charts show the maximum corrosion rate expected over a wide range of hydrogen sulfide concentration and temperature likely to be encountered in petroleum catalytic refining processes utilizing hydrogen. From a practical standpoint, these charts are in agreement with the results reported by other investigators.

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DISCUSSION

Comments by A. Dravnieks, Standard Oil Company, Whiting, Indiana

The plots presented fit satisfactorily, in the upper part of the field, into the general field of various similar plots which have been published previously. However, in the lower right part of the field, the curves follow the dangerous path of ignoring thermodynamics and, in our opinion, are basically wrong. Correlations of numerous literature data, of our own published experimental work and of more recent work by Prof. Delahay, show that whereas hydrogen acts substantially as an inert diluent at high H_2S concentrations, this is not the case at low concentrations. For this reason, it is basically incorrect to ignore hydrogen as a thermodynamic factor at low hydrogen sulfide-hydrogen ratios.

It is important to remember that at low hydrogen sulfide concentrations in the presence of hydrogen and hydrocarbons, there are three concentrations which must be considered. In simple terms, hydrogen sulfide tends to produce iron sulfide scale; hydrogen tends to reduce the sulfide scale back to iron. At a certain ratio of hydrogen sulfide to hydrogen, both tendencies balance and neither reaction takes place; no corrosion occurs at or below this ratio. This effect has been well established both in laboratory tests and in plant operations. Slightly above the critical ratio, corrosion will be slow. In this range a third component, such as hydrocarbon vapor, can dilute the hydrogen sulfide—hydrogen mixture still further, and can slow down, but not stop, the corrosion.

We criticize the plot presented on two points:

1. Hydrogen sulfide concentration alone, in the low concentration range, does not define the corrosion rate uniquely, as the plots presented tend to indicate. For instance, at 1000 F and at the 0.02 percent H_2S level, with the rest of gas principally hydrogen alone, corrosion would not occur; rather, iron sulfide would be reduced to iron, as has been observed in the laboratory, and in the plant when the stream contains approximately 70 percent H_2, with the remainder hydrocarbons. However, if the amount of hydrogen in the gas is only 2 percent, again at the 0.02 percent H_2S level, and the remaining 97.98 volume percent is hydrocarbons, corrosion at 1000 F becomes thermodynamically possible and would occur at a rate of 100-200 mils per year. One sees that, at a constant low hydrogen sulfide level, corrosion rate can vary greatly depending on the composition of the remaining gas. Thus, plots on a concentration basis are applicable only to a limited range of overall gas composition, and one should be very careful not to generalize the data. We propose that if plots are given, on a concentration basis, the composition of the remainder of the gas should always be clearly defined.

2. Although the general shape of the iso-corrosion lines in the plot suggests that the thermodynamic equilibrium line would be encountered in the field somewhere in the lower right corner, the iso-corrosion lines cut across the equilibrium line which was established by Rosenqvist and other workers and substantiated also by our published work.* We assume that the mixtures of gas used for the preparation of the plot contained only H_2S and H_2. Figure 3 indicates the approximate position of the generally accepted equilibrium line with respect to the presented series of iso-corrosion lines. It is apparent

* *Proceedings of API*, 37, III, 100 (1957); *J. Electrochem. Soc.*, 105, 183 (1958) Apr.

that corrosion rates of appreciable magnitude have been found in the range where other workers have observed the reduction of sulfides to metal. This leads us to believe that either oxygen has been present, causing the oxygen effect which has been discussed in the footnoted references, or else the determinations of low concentrations of hydrogen sulfide did not represent correctly the true concentrations.

We found in our work that it is difficult to obtain reliable low level hydrogen sulfide determinations in hydrogen gas unless certain techniques are applied; these are described in the second of the above references. If the hydrogen sulfide concentrations determined experimentally were too low by certain incremental values, a cross-over with respect to the thermodynamic equilibrium line would be observed, as seems to be the case. Of course, as long as the same method of analysis is used, iso-corrosion plots still would be useful as a practical guide for adjusting reforming operations. However, if the source of the discrepancy between these and other data is indeed in the analytical determinations, the curves become somewhat arbitrary and too dependent on the variation of analytical techniques to be universally useful in the lower right corner of the plot.

Reply by E. B. Backensto:

We appreciate the remarks by Dr. Dravnieks which deal largely with the theoretical aspects of high-temperature corrosion in a hydrogen atmosphere. However, a discussion of theory was beyond the scope of this paper. The present paper was prepared primarily to make available the iso-corrosion rate curves presently being used by our company. These curves represent an extension and revision of our previously published information.

As we understand it, Dr. Dravnieks raised the following main objectives: thermodynamics were ignored, using H_2S concentration as a parameter without qualification is incorrect, oxygen may have been present in our tests, and the accuracy of our H_2S determinations was questioned. We shall try to clarify our position on these points.

The fact that our curves dip slightly below the thermodynamic cut-off has been mentioned previously. Our study, however, did not include fundamental aspects which might verify or contradict thermodynamics. We were interested in obtaining practical information which could be used by design and operating people. Since we have been using these laboratory data for some time, as mentioned in the paper, we have been able to check the results numerous times by test coupons exposed in field units. In all cases, except as mentioned in a previous paper,[5] we feel that the results of laboratory tests are in agreement with those obtained in commercial units.

We have recognized the fact that at low H_2S concentrations the corrosion reaction theoretically stops. This principle is being applied for the reduction of corrosion in catalytic reformers by the use of naphtha pretreaters or desulfurizers.[4, 5] It might be pointed out that measureable corrosion was found in a commercial reformer operating below the thermodynamic cut-off.[5]

We have attempted to present a summary of laboratory results in the most simple and usable form. We feel that the major parameters are H_2S concentration and temperature. No assumption is necessary that laboratory results were obtained in a hydrogen stream containing the indicated amount of H_2S. This point was made in our first publication on this subject.[2] We feel that as long as the gas stream contains more than 10-20 percent vol hydrogen, the curves presented will apply. This, we believe, represents most field conditions and, from a practical standpoint, no complicated qualifications are required.

In our laboratory work, the hydrogen used was passed through a De-oxo unit and drier prior to entering the test chamber. The few tests made indicated the absence of oxygen in our hydrogen stream.

We used an adaptation of the Tutwiler analysis in the laboratory for H_2S content of the gas. We believe that this is the simplest method of analysis, capable of producing as accurate a result, particularly for low H_2S concentrations, as any other routine procedure available. We also recommend that this method be used for field testing.

The Evaluation of Certain Organic Nitrogen Compounds As Corrosion Inhibitors*

By E. J. SCHWOEGLER* and L. U. BERMAN*

Abstract

The Static Water Drop Test adapted to research methods has been utilized to evaluate the corrosion inhibiting ability of a number of heterocyclic nitrogen compounds. Most of these heterocyclic N derivatives were synthesized and purified and evaluated in a petroleum-type lubricating oil and a synthetic diester-type lubricating oil.

The effect of various functional groups and their positions in the molecule was related to the corrosion inhibiting properties. It was found that the adsorptive power was a function of the heterocyclic nitrogen containing a hydrogen atom as the substituent. It was also found that the adsorptive power at the heterocyclic nitrogen atom was lost when other substituents replaced the hydrogen. It was concluded that the adsorptive effect of the heterocyclic nitrogen is due to its unshared electron pair, which is sterically hindered when substituents other than hydrogen are attached.

N-Hydroxyalkyl derivatives in diester oil seemed to arrange themselves in order of effectiveness on the basis of the position of the hydroxyl group in the alkyl chain. The closer the hydroxyl was to the heterocyclic nitrogen the more effective it was as a corrosion inhibitor. There was no way to explain the order of effectiveness of the same compounds in petroleum oil. 5.8.1

Introduction

THE STATIC Water Drop Test developed by Baker, Jones, and Zisman[1] and adapted to research methods by Schwoegler and Berman[2] was utilized to study certain heterocyclic nitrogen compounds as corrosion inhibitors in a synthetic ester and a hydrocarbon lubricant.

The study was directed primarily to determining the effect of N-substituted morpholines on inhibition. Piperidine and 2,5-dimethyl piperazine were also included for comparison. The compounds used in the study were either synthesized in the Armour Research Foundation laboratories or obtained from various commercial sources.

Experimental Work

The synthetic lubricant used in this study was an activated carbon-treated dioctyl sebacate (Plexol 201). It had a flash point of 430 F and a solidification point of —70 F. The kinematic viscosity was 12.5 centistokes at 100 F and 1450 centistokes at —40 F. The hydrocarbon lubricant was the petroleum oil which constituted the base material for the AN-06a specification. It had a flash point of approximately 290 F and a pour point of —75 F. The kinematic viscosity was 10.6 centistokes at 100 F and 2900 centistokes at —40 F. The test fluid was prepared by weighing an indicated quantity of an organic nitrogen compound into one of the respective lubricants.

The metal test specimen was a triangularly shaped SAE 1020 steel specimen with a smooth, spherical, shallow depression approximately 0.1 inch deep. It was prepared and polished by Schwoegler and Berman's procedure.[2]

The polished metal test specimen was placed in a 100-ml beaker, and test fluid was added to the beaker until it was 3-5 mm above the top of the metal specimen. The beaker was covered with a dry watch glass and placed in a forced air oven held at 140 ± 1 F for a 1-hour period. At the end of this time the test unit (covered beaker and contents) was removed, and 0.2 ml of carbon dioxide-free distilled water was introduced below the surface of the oil into the dimpled area of the metal test specimen. The test unit was returned to the oven and the corrosion noted at the end of a 48-hour period. Corrosion was recorded as a function of the area covered by the water drop. A 0-10 scale was used, a reading of 10 indicating that 100 percent of the area had been corroded. A corroded area of less than 10 percent (1 scale reading) was recorded as a fraction of 1. Therefore, a reading of ½ meant a 5 percent corroded area. All evaluations were made in duplicate.

The organic compounds evaluated in this study had purities of 95 percent or better and most were at least 99 percent pure materials. The compounds obtained from commercial sources were fractionated and a center cut was taken, or they were recrystallized before evaluation. A number of compounds were synthesized in the Armour Research Foundation laboratories. The following N-substituted morpholines were prepared by either the method described by Leffler and Volwiler[3] or a slight modification of it: N-n-butyl, N-phenylpropyl, N-2-hydroxypropyl, N-3-hydroxypropyl, N-4-hydroxybutyl, N-2-methoxyethyl, and N-2-ethoxyethyl morpholine and 1,2-di-N,N′-dimorpholinoethane. In this method an access of morpholine is reacted with the corresponding halide by refluxing in a benzene solution. 4,5-Dimorpholinocatechol was prepared by the method of Henry and Dehn,[4] in which morpholine and catechol in the molar ratio of 2 to 1 are reacted by refluxing for 12 hours in

TABLE 1—Corrosion Inhibition by Certain Organic Nitrogen Compounds in Diester and Petroleum Lubricants for a 48-Hour Test Period

Compound	%	CORROSION RATING	
		In Diester	In Hydrocarbon
Morpholine	0.2	0	1/8
	.1	0	1/2
	.05	0	
	.01	10	
N-Methyl morpholine	.2	10	10
N-n-Butyl morpholine	.2	10	10
N-Phenyl morpholine	.2	10	10
N-3-Phenylpropyl morpholine	.2	10	10
N-Aminoethyl morpholine	.2	1/8	1/32
	.1	1/8	
	.075	1	
	.05	2	
	.025	10	
	.01	10	
N-Dimethylaminoethyl morpholine	.2	0	1
	.02	1	
N-Diethylaminoethyl morpholine	.2	0	2
N-Aminopropyl morpholine	.2	0	0
	.1	0	0
N-Hydroxyethyl morpholine	.2	0	4
	.1	1/8	
	.05	4	
N-2-Methoxyethyl morpholine	.2	1/8	2
N-2-Ethoxyethyl morpholine	.2	10	9
N-2-Hydroxypropyl morpholine	.2	0	2
	.1	1/8	6
N-3-Hydroxypropyl morpholine	.2	1/32	3
	.1	7	
N-4-Hydroxybutyl morpholine	.2	1/32	3/4
1, 2-Di-N, N′-dimorpholinoethane	.2	1	8
4, 4′-Dithiomorpholine	.2	10	10
4, 5-Dimorpholinocatechol	.2	10	10
2, 4, 6-Trimorpholinomethyl phenol	.2	10	10
N-2-(2-Ethylhexamido)ethyl morpholine	.2	10	10
Piperidine	.2	0	0
2, 5-Dimethyl piperazine	.2	0	0
	.1	0	
	.05	0	
	0.01	0	

★ Submitted for publication April 3, 1958. A paper presented at the Fourteenth Annual Conference, National Association of Corrosion Engineers, San Francisco, California, March 17-21, 1958.

* Armour Research Foundation of Illinois Institute of Technology, Chicago, Illinois.

ethanol in the presence of air. 2,4,6-Trimorpholinomethyl phenol was prepared through the Mannich reaction as described by Bruson and MacMullen.[5] N-2-(2-Ethylhexamido)ethyl morpholine was prepared by reacting 2-ethylhexoic acid with N-aminoethyl morpholine.

The results obtained with the various organic nitrogen compounds in the synthetic diester and the petroleum base oils are given in Table 1.

Discussion of Results

Morpholine itself was found to be an effective inhibitor in both oils, although it responded slightly better in the diester oil. Substitution of the oxygen atom in morpholine by $-CH_2-$, as in piperidine, maintained the effectiveness in the diester and actually improved the inhibition in the petroleum oil. Similarly, substitution of the oxygen atom by -NH-, as in 2,5-dimethyl piperazine, did not diminish the corrosion inhibiting characteristics. In fact, the data for the diester oil show that 2,5-dimethyl piperazine was substantially more effective than the other two compounds.

The effectiveness of certain polar organic compounds as corrosion inhibitors has been shown by many investigations to be due to the ability of these materials to be preferentially adsorbed at the metal surface. The corrosion inhibiting properties are directly related to the adsorbability of the polar material and the amount adsorbed. In the Static Water Drop Test the adsorption takes place before the water drop is introduced into the dimpled area. The polar compound forms an adsorbed film and the hydrophobic nature of this film is enhanced by the vehicle, in this study either a petroleum or a synthetic diester lubricant. This film prevents the water from reacting with the metal. If the inhibitor is fairly soluble in water, as were the morpholines and the other compounds studied, inhibition will not be impaired when there is sufficient inhibitor present to maintain an effective barrier layer in spite of the displacement and dissolution effects of the water.

This adsorption phenomenon explains the effectiveness of morpholine, piperidine, and 2,5-dimethyl piperazine in both oils. Morpholine and piperidine probably adsorb at the heterocyclic nitrogen, and thus the molecules extend outward from the surface of the metal. 2,5-Dimethyl piperazine has two points of adsorption, at the two heterocyclic nitrogens. Thus, it is probably adsorbed in the plane of the metal surface. The molecule covers a larger surface area and therefore smaller quantities are required for effective inhibition. This idea was postulated by Mann[6] to account for the superior corrosion inhibiting properties of other organic nitrogen compounds.

The enhanced effectiveness of 2,5-dimethyl piperazine might also be due in part to the presence of the methyl groups, which are ortho to the heterocyclic nitrogen. Mann[6] observed similar effects with toluidines and Rhodes and Kuhn[7] had corresponding results with the pyridine-lutidine-picoline-collidine

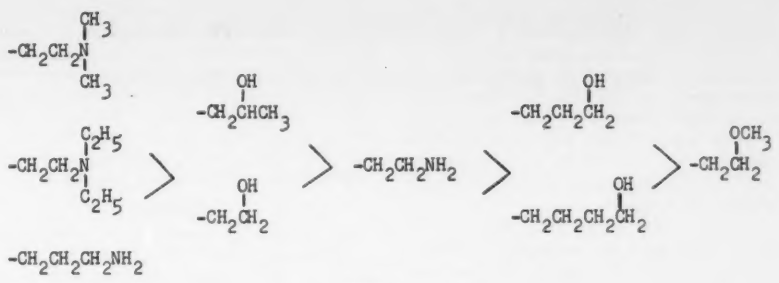

Figure 1

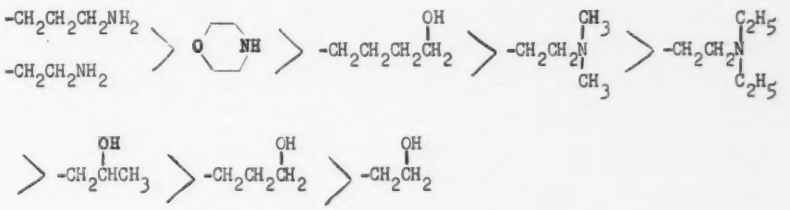

Figure 2

series. This effect was later described as the ortho effect by Cardwell and Eilers.[8]

When the hydrogen of the heterocyclic nitrogen was replaced by an alkyl, aryl, or aralkyl group, the corrosion inhibiting characteristics of the morpholine compound were destroyed. N-Methyl, N-n-butyl, N-phenyl, and N-3-phenylpropyl morpholine exhibited no inhibiting properties in either oil.

Similar results were obtained when the hydrogen was substituted by sulfur, as in 4,4′-dithiomorpholine. Substitution of the hydrogen by ethylene, as in 1,2-di-N,N′-dimorpholinoethane, substantially eliminated corrosion inhibition in the petroleum oil and reduced it somewhat in the diester. The different results observed with the latter compound could be due at least in part to differences in the two oils. This was noted to a greater degree for other morpholine derivatives which are discussed later.

Substitution of the hydrogen of the heterocyclic nitrogen by polar substituents, such as amino and hydroxyl, did not destroy the adsorptive power of the morpholine, and hence these derivatives possessed corrosion inhibiting characteristics in varying degrees. Substitution by phenolic hydroxyl-containing groups did destroy the adsorptive power.

N-Dimethylaminoethyl, N-diethylaminoethyl, N-aminopropyl, N-2-hydroxypropyl, N-hydroxyethyl, N-aminoethyl, N-3-hydroxypropyl, N-4-hydroxybutyl, and N-2-methoxyethyl morpholine were slightly less effective than morpholine itself in the diester oil. The series showed slightly decreasing effectiveness in the diester oil as shown in Figure 1.

Amino substitutents, in general, were slightly more effective than hydroxyl substituents or their derivatives. No order of effectiveness was evident for the amines, but inhibition by the hydroxyl compounds appeared to be related to the distance of the hydroxyl group in the chain from the heterocyclic nitrogen.

In petroleum oil, the same substituted morpholines exhibited a somewhat different behavior both in relation to morpholine and in relation to each other. This difference can be attributed to the difference in oils. The N-aminopropyl derivative was found to be as effective at 0.2 percent concentration as piperidine and 2,5-dimethyl piperazine. The N-aminoethyl derivative was less effective, but both derivatives were slightly better than morpholine itself. N-dimethylaminoethyl was somewhat more effective than N-diethylaminoethyl morpholine, and both were less effective than morpholine. The behavior of the hydroxyl-containing substituents and their derivatives could not be explained by the hydroxyl position, as in the diester oil. The N-4-hydroxybutyl derivative, which was substantially as effective as N-dimethylaminoethyl morpholine, was the most effective of the hydroxyl-containing substituents. It was followed in order by the N-2-hydroxypropyl, N-2-methoxyethyl, N-3-hydroxypropyl, and N-hydroxyethyl derivatives.

These substituents arranged themselves in the activity series in the petroleum fluid as shown in Figure 2.

The N-ethyoxyethyl derivative showed no inhibiting properties in either oil.

When morpholine was substituted at the heterocyclic nitrogen on a phenolic ring, as in 4,5-dimorpholinocatechol, or to a phenolic ring through a methylene group to the nitrogen, as in 2,4,6-trimorpholinomethyl phenol, the inhibiting properties also were lost.

In general, amino substituents were found to be more effective than hydroxyl-containing substituents. This is in accord with the results obtained by Zisman,[9] who showed amines and acids to be more readily adsorbed than other polar com-

pounds such as alcohols, esters, ketones, and phenols.

The results of this study indicate that substitution of organic groups for the hydrogen of the heterocyclic nitrogen of morpholine destroys the adsorptive power at this point. This was concluded from the results obtained with N-methyl, N-n-butyl, N-phenyl, and N-3-propylphenyl morpholine.

It was also concluded that due to steric hindrance the adsorptive power at the heterocyclic nitrogen is also lost when the hydrogen is substituted by other polar-containing groups. However, adsorption of this type of compound does take place at the polar group of the substituent. In this regard it was assumed that the adsorption of morpholine is not at the hydrogen on the heterocyclic nitrogen but occurs at the unshared electron pair of the nitrogen. The hydrogen of the morpholine nitrogen does not interfere with adsorption at the point of the unshared electrons, but other groups do sterically hinder the adsorptive power.

It was further concluded that the rigid position of the ring nitrogen so fixes the unshared electrons that steric effects are quite pronounced when substituents other than hydrogen are present. This rigid condition is not present in amino-containing substituents, hence adsorptive power is still possessed by the amino nitrogen of the substituent. This is true even when the two hydrogens of the amino group are substituted by alkyl groups such as methyl or ethyl as in N-dimethylaminoalkyl and N-diethylaminoethyl morpholine. In this instance the unshared electron pairs apparently are still available for adsorption. Certain limited steric effects would undoubtedly alter the effectiveness of these materials. Conversion of N-aminoethyl morpholine to an amide by reaction with 2-ethylhexoic acid resulted in a product with little or no adsorptive power. This was expected since the electronic character of the nitrogen in amides is radically different from that of the nitrogen in amines. The electron pair is no longer available due to internal resonance.

Summary

A number of heterocyclic nitrogen compounds, mostly N-substituted morpholine derivatives, were evaluated in a petroleum lubricant and a synthetic diester lubricant by the Static Water Drop Test. The results indicated that the adsorptive power is a function of the heterocyclic nitrogen containing a hydrogen atom as the substituent. It was also shown that the adsorptive power at the heterocyclic nitrogen atom is lost when other substituents replace the hydrogen. Adsorption noted with substituents containing polar amino and alkyl hydroxyl groups occurs at the polar group of the substituent. The latter adsorption was evidenced by the ability of these compounds to function as corrosion inhibitors. Substituents with phenolic hydroxyl and amide linkages or ones such as sulfur and ethylene which link two morpholines seemed to have limited adsorptive power or none at all.

It was concluded that the adsorptive effect of the heterocyclic nitrogen is due to its unshared electron pair, which is sterically hindered when substituents other than hydrogen are attached. The steric effect is influenced by the fixed position of the heterocyclic nitrogen in the ring. When aliphatic nitrogen atoms such as those encountered in the alkyl amino morpholines are in the group replacing the hydrogen, adsorption apparently occurs at the unshared electrons of the aliphatic nitrogen. A more flexible situation exists here, which allows the unshared electrons to be available for adsorption even when the alkyl amino group is further substituted with methyl and ethyl groups as in N-dimethylaminoethyl and N-diethylaminoethyl morpholine.

N-Hydroxyalkyl derivatives in diester oil seemed to arrange themselves in order of effectiveness on the basis of the position of the hydroxyl group in the alkyl chain. The closer the hydroxyl was to the heterocyclic nitrogen the more effective it was as a corrosion inhibitor. There was no way to explain the order of effectiveness of the same compounds in petroleum oil.

Acknowledgments

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Any discussions of this article not published above will appear in the June, 1959 issue

Corrosion Inhibitor Testing Inside a Products Pipe Line*

By ROBERT H. MEYER

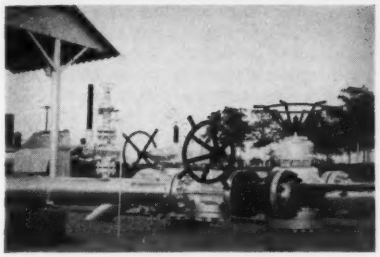

Figure 1—View of pipe manifold showing special test segment of pipe and corrosion coupon installation apparatus. Special test segment of pipe in dark color to right of center is arranged to allow removal without shutting pipe line down. Corrosion coupon installation device is in left center of picture.

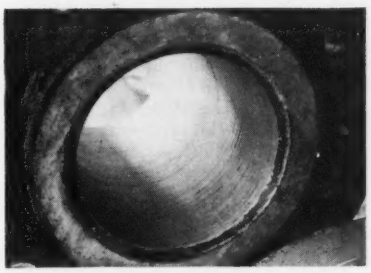

Figure 2—Internal view of special test segment of pipe. This view shows portion of internal surface after 35 days exposure to refined petroleum products containing Inhibitor Y.

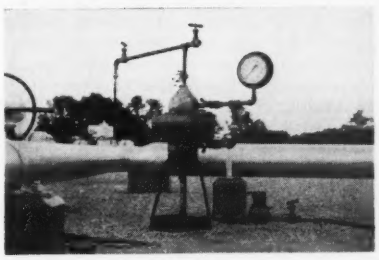

Figure 3—Analytical type high pressure filter device. Portion of product flowing in pipe is sampled from center of pipe, filtered through No. 42 Whatman filter paper in filter device, and volume is measured by positive displacement meter. Gauge is for use in determining differential pressure across filter paper and support.

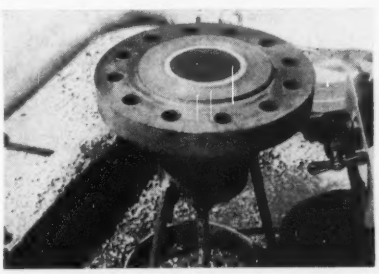

Figure 4—Analytical type high pressure filter device in open position showing exposed 12.5 cm Whatman No. 42 filter paper after use. Paper is supported by ⅛ inch thick porous stainless steel disk.

TABLE 1—Inhibitor Dosages

Inhibitor	Dosage Lbs/M Bbl.	Length of Test, Days
X_1	6	42
X_2	9	43
Y	8	51
Z	6	61

Abstract

Observations and data obtained during the testing of four different oil soluble corrosion inhibitors in a 151 mile section of a products pipe line are discussed in detail. Seven evaluation procedures were used. These procedures were based on use of data from: (1) Line test coupons, (2) Specially prepared test segments of pipe, (3) Product filtration, (4) scraper trap deposits, (5) frequency of strainer plugging, (6) modified turbine type rust tests, and (7) "C" factor or roughness factor. Evaluation procedures four and five are relative, since they are especially dependent upon changes in operating conditions. The test coupon method of evaluation is the most desirable because of its simplicity and reliability.

A very slight depletion of inhibitor in the product was generally indicated after it had been transported 151 miles even though a high degree of overall protection was maintained.

One of the inhibitors tested produced a corrosion product of a bulky and amorphous structure which caused accelerated strainer plugging even though the overall rate of corrosion was low. X-ray diffraction analyses of the material deposited on the filters was found to be a method of determining the relative amount of corrosion products formed in the pipe line during tests of the various inhibitors. 5.8.1

Introduction

OIL SOLUBLE corrosion inhibitors were first used in refined petroleum products pipe lines some 10 years ago. With the increased use of oil soluble corrosion inhibitors, the number of commercially available inhibitors of that type also increased. Laboratory tests indicated that many of the new inhibitors were good, but an actual pipe line comparison and test was desired to augment laboratory data. The desire to test a new inhibitor, Sinclair RD-155, and to compare it to the inhibitor being used, Sinclair RD-119, led to a testing program.

The selection of a test location was based on a number of factors. A location was needed where products could be received that contained no inhibitor, so that inhibitors could be injected under control. A location was needed that afforded a backlog of information on the inhibitor that was currently being used. A section of pipe line located in the least changeable climate was necessary to minimize the effect of temperature, when comparing results from testing through different seasons of the year. In order to obtain a good comparison, it was necessary that the section of pipe line chosen be clean with its internal surfaces under good corrosion control. If possible, a portion of pipe line was required that would provide facilities at a point about half way along the section chosen for the test. Using these facilities a comparison could be made between products having traversed half the test section and those that had been pumped through all of it. Since the most severely corrosive condition in a products pipe line occurs when the product is static, it was preferred to use a portion of pipe line that was not operated all the time.

The location best fulfilling these conditions, and thus chosen for the tests was the Corpus Christi (Nueces Station) to Luling, Texas, section of the Corpus Christi, Austin, San Antonio system. This is a section of eight-inch pipe line, 151 miles long, consisting of a section of new pipe and a section of converted crude line. The portion from Nueces Station to Victoria is 76 miles long while the section from Victoria to Luling is 75 miles long.

This line was built and placed in operation in 1948 and provided a good background from the internal corrosion standpoint. Only kerosene and two grades of gasoline were handled in this pipe line. All shipments, since this line was placed in operation, have contained an effective corrosion inhibitor which was blended in them at the refinery. As a result the line was relatively smooth and free of rust.

★Submitted for publication January 27, 1958. A paper presented at a meeting of the North Central Region, National Association of Corrosion Engineers, Chicago, Illinois, October 2, 1957.

General Details

During the test period, uninhibited products were received from the refinery. At Nueces Station, storage facilities were made available for the inhibitors to be tested. The inhibitors were continuously injected in the proper amounts by a chemical proportioning pump into the stream received from the refinery. Only oil soluble inhibitors were chosen for the test program because of certain operational limitations preventing the use of water soluble inhibitors. Injection of another inhibitor started immediately after completion of each test. As a result no uninhibited products entered the line between tests. This procedure maintained a high degree of corrosion control but resulted in a period after each test during which the control was the result of both inhibitors. Two weeks operation was allowed on each inhibitor, before data were collected, to minimize the effects of the inhibitor previously used.

In the operation of a pipe line, scrapers are used to keep the internal surfaces of the pipe free of foreign matter and to remove as much corrosion product and mill scale as possible. Prior to these experiments, scrapers were run about three times a year. During the test period, however, a scraper and brush were run through the line after each inhibitor had been tested. This removed as much of the remaining inhibitor as possible, and ensured the same internal condition for the subsequent inhibitor. All scrapers put

About the Author

ROBERT H. MEYER—Project engineer for Sinclair Pipe Line Company, Independence, Kansas. He is now in charge of liaison with Sinclair Research Laboratories, Inc. on pipe line research and development projects. He has a BS in chemical engineering (1949) Kansas State College and was with Phillips Petroleum Co. for a year. For two years he was with the Oil Movement Dept. of Sinclair and since then a member of the engineering department. He is a member of NACE.

into the line were completely reconditioned and in as nearly the same condition as possible.

Unavoidable variation of operation during the test period was held to a minimum. An average of 9,200 barrels per day entered the line at an average of 75-80 F and was withdrawn from the line at 70-73 F. The line was operated fifteen hours a day, five days a week. Eighty hours of pumping at that rate were required to displace the line.

The test program was initiated by obtaining additional data on the inhibitor that had been used ever since the line was put into service. Since this inhibitor was very satisfactory, it was used as a basis of comparison for the other inhibitors.

Following fifteen days of tests on the regular inhibitor, Sinclair RD-119, the other inhibitors were tested in the following sequence, and for the periods shown in Table 1. The dosage was determined by laboratory tests to be the quantity required to maintain the same level of corrosion control as RD-119.

Evaluation Procedures

Line Test Coupons

Polished steel test coupons, approximately 4½-inch x ½-inch x ⅛-inch, were installed in the stream at Nueces and Luling Stations. The preparation and handling of these coupons is critical if consistent and reliable results are to be obtained. Discrepancies among tests were minimized by consistent use of cold-rolled SAE 1020 steel, polished to a standardized roughness of 35 microns.[1] One hundred-grit silicon carbide cloth belts were used to obtain this roughness. During grinding, coupons were held by powerful permanent magnets to prevent moist finger prints from damaging the surface.

Following surface preparation, the coupons were individually packed in an envelope treated with a volatile corrosion inhibitor. The coupons were installed through a special device which could be used without shutting the line down. This device consisted of a special two-inch pipe line tap, valve and stuffing box.[2]

The test coupons were graded on a visual basis, since weight losses were frequently small compared to losses resulting from wearing or peening while in the pipe line. The coupons were rated by percent of area corroded, as shown in Table 2 and by intensity of corrosion.

The results of the various inhibitors on in-line test coupons are shown in Table 3.

TABLE 2—Percent of Coupon Area Corroded

Rating	Percent Surface Corroded
A	None
B++	Less than 0.25
B+	0.25 to 5
B	5 to 25
C	25 to 50
D	50 to 75
E	75 to 100

Specially Prepared Test Segments of Pipe

A one foot long segment of pipe or flanged spool was used for a visual test. This test segment was installed in a portion of the Luling Station manifold in such a manner that it could be removed for periodic visual inspection without disrupting the operation of the pipe line. Figure 1 illustrates the way the segment was installed in a portion of the above ground manifold.

The test joint was prepared with a brightly machined inside surface completely free of corrosion. A new spool was inserted at the beginning of each test. Table 4 shows the data from these test segments. The method of visual rating of the spools is the same as that described in the line coupon test description, and ratings are the same as shown in Table 2. The spools are rated by each quadrant. The degree of corrosion of the pipe segment is much more severe than for the line test coupon since the line coupons did not have as rough a surface as the pipe segments. Evans[3] as well as King and Rau[4] have found that on ground surfaces corrosion occurs first on the major abrasion lines. Since a rougher surface would have more abrasion lines, the higher corrosion rates on the test segment would be expected.

Inhibitor Y was superior to the other inhibitors in this test, with only 5 percent of the surface rusted after 35 days exposure. A view of part of the internal surface of this segment is shown in Figure 2. All other inhibitors allowed a much greater amount of corrosion, with inhibitor X_1 permitting an average of 53 percent corroded area in only nine days exposure. One spool exposed to inhibitor Z for 47 days had only 31 percent of the area corroded. The test spool was re-machined, returned to the line, exposed to inhibitor Z for 11 days, and then inspected again. It then exhibited 46 percent corroded area. This could indicate that the major portion of the rust occurs during the first few days exposure, or that the degree of protection given by inhibitor Z was declining as the test progressed. Inhibitor Z did follow inhibitor Y which showed such excellent results. The results obtained, therefore, during the 47 day test may have been due to small residual amounts of inhibitor Y remaining in the pipe system, thus carrying over into the subsequent test.

It was noted that inhibitors X_1 and Y permitted considerably more corrosion at the bottom of the line than at the top. The rust streaks on the bottom of the

TABLE 3—Ratings of Inhibitors on In-Line Test Coupons

Inhibitor	LOCATION			
	Nueces		Luling	
	Rating	Days Exposed	Rating	Days Exposed
W	B+ (2%)	31	A	31
X_1 (low dosage)	A	13	B (15%)	22[1]
X_2 (high dosage)	B+ (2%)	32	B+ (1½%)	27
Y	A	23	A	26
Z	E (100% Heavy)	26	B+ (¾%)	28

[1] Coupon had "A" rating after 9 days exposure.

TABLE 4—Pipe Segment Corrosion Rating Data[1]

Inhibitor	Days	QUADRANT				Average Rusted Area
		First[2]	Second	Third[3]	Fourth	
W, X_1	44[4]	80% medium + 10% light	40% medium + 40% light	30% light	30% light	58%
X_1	9	60% medium to heavy	50% medium to heavy	50% medium to heavy	50% medium to heavy	53%
X_2	40	33% light to medium	30% light	30% light	30% light to medium	31%
Y	35	3% light to medium	5% medium	10% light to medium	2% light	5%
Z	47	55% medium to heavy	37% medium	15% light	15% medium	31%
Z	11	72% medium to heavy	35% medium	32% light	45% medium	46%

[1] Test segment in line at Luling.
[2] Bottom quadrant.
[3] Top quadrant.
[4] Twenty two days on Product W, twenty two days on Product X_1. Eight days exposure to Inhibitor W gave a B+ (0.25 to 5%) rating.

pipe are probably the result of moisture and other foreign material that settled out of the products during static periods. Inhibitor Y and X_2 therefore appear to provide protection under static conditions.

Product Filtration

A special high pressure, analytical type filter device was fabricated to provide measurement of suspended solids passing through a portion of the line at a given time. Whatman No. 42 analytical filter papers 12.5 centimeters in diameter supported on a porous stainless steel disk were used as filter media. These filter papers were very carefully dried and weighed before and after use. The amount of product filtered was measured by means of a positive displacement meter. (The meter was calibrated according to API Code 1101.) This allowed the weight of sediment per unit volume to be calculated. Figure 3 shows the filter device in actual use while Figure 4 shows the device opened to expose the filter paper on the porous media after use.

Filtration tests of products received at Nueces Station indicated that the amount of solids per barrel remained constant while products were being pumped from a single tank, even though a greater amount of solids was expected from liquids in the bottom of a tank than from liquid near the top. The amount of entrained solids was therefore quite consistent throughout each tender received. Samples from the same portion of a batch or tender of product were filtered successively at Nueces, Victoria, and Luling. The data prior to and immediately after scraper runs were taken in an effort to keep conditions as consistent as possible (see Table 5).

A study of the mass of data obtained at specific time intervals prior to and immediately following scraper runs indicated that filtration data at those times is especially valuable. The data obtained before a scraper run indicate by the variation in the amount of solids removed the apparent amount of depletion of a specific inhibitor. The data obtained following a normal scraper run where inhibitor efficiency was good, show that the quantity of suspended solids dropped rapidly after a short period of time and after four hours was almost down to the same quantity suspended before the scraper run. In cases where poor inhibitor efficiency was exhibited, the correlation was observed that the length of time that the quantity of suspended solids remained high was much longer. In addition, the total time required to return to a normal level was much longer than with inhibitors exhibiting good over-all efficiencies. Analysis of the sediment removed following a given scraper run indicated that the increase in solids occurring as a consequence of using an inefficient inhibitor was largely due to the formation of a new rust in the pipe line while that inhibitor was being tested. Even though scraper runs may vary greatly under the best controlled conditions, the variation in sediment removed was not great enough in magnitude in this series of tests to prevent the correlation described above.

The gain in solids carried by the stream during tests of inhibitor Y, RD-155, was very low compared with other inhibitors. Since the gain in the latter half of the line was less than that in the first half of the line, the inhibitor depletion, if any, was not very rapid. The coarseness of the solid material was a definite factor in the quantity of product that could be filtered, and varied considerably with inhibitors.

The amounts of solids carried in the stream after a scraper run dropped off rapidly with inhibitors W, X_1, Y and Z. However, with X_2, the solids were very fine and were carried in the stream much longer. The decrease was gradual, requiring five hours to reach a level the others reached in a much shorter time. For that reason, the data shown on Table 5 for inhibitor X_2 prior to passage of the scraper require much less interpretation than those after the scraper run.

Scraper Trap Deposits

At the end of each inhibitor test period, a knife and a brush scraper were run one hour apart. Solid material removed from the scraper trap screens at Luling Station after the scrapers had traversed 151 miles of pipe were weighed and analyzed (see Table 6).

The amounts of mill scale and rust were determined by a semi-quantitative X-ray diffraction technique. The higher the quantity of mill scale the darker the color of the sample. This is attributed to the very dark color of mill scale and the light yellowish-brown color of ferric oxide hydrates. Mill scale, composed of large crystalline Fe_3O_4 and FeO, is formed on the steel during manufacture, and various amounts are scraped from the line depending on the efficiency of the scrapers. The material identified as rust in Table 6 is hydrated ferric oxide (FeOOH or $Fe_2O_3 \cdot H_2O$), which is a typical corrosion product resulting from the presence of oxygen and water. Anhydrous Fe_2O_3 is rarely found in corrosion products formed in the presence of moisture, and none was found in any of the samples taken during the course of the test program.

Analyses of material removed from the scraper trap indicated that inhibitor Y permitted the least amount of corrosion product to be formed, even though more material was removed by the scraper run following use of inhibitor Y than for any of the other scraper runs. The large amount of mill scale undoubtedly resulted from an unusually effective scraper run. Although the amount of solids collected near the end of the test on inhibitor Z was small, considerable amounts of solids were collected and removed by the filter screens at Luling throughout most of the time that this inhibitor was in use. The data on quantity of rust removed indicate that inhibitor Y gave substantially better protection at the dosages tested than did the other inhibitors.

Frequency of Strainer Plugging

Frequency with which filter screens plugged was another indicator of inhibitor efficiency, and measured the amount of loose corrosion products formed in the line. During normal operations, all products entering the meters at Luling Station were filtered through 60 mesh screens. In normal operation with inhibitor W, these screens required cleaning about once a month. The product ordinarily removed was a very dark granular solid.

Red powdery rust was formed during use of inhibitor X_1 and was filtered out of the products by strainer screens. This necessitated cleaning the screens every day when the inhibitor had had time to take full effect.

With inhibitor X_2, which was the same material as X_1 but in a higher concentration, the rate of plugging decreased to about once per month. The solid material on the screens was very dark in color.

The screens did not require cleaning during the entire test of inhibitor Y. At the end of 30 days, a routine check was made to determine that the screens were not broken, but cleaning was not required at that check.

With inhibitor Z, the frequency of plugging the strainers increased markedly from about once a month to four times a day by the end of the test. The solid material removed was reddish-brown in color and tended to cake on the screens.

TABLE 5—Amount of Solids Before and After Scraper Run

Inhibitor	Before or After Scraper Run	Average Lbs of Solids Per 1000 bbls of Products		
		Nueces[1]	Victoria	Luling
W.....	Before	.07	.22	1.04
X_1.....	Before	.04	.23	1.42
	After	.04	.41	1.77
X_2.....	Before	.30	.34	1.36
	After	.06	1.38	2.18
Y.....	Before	.19	...	.14
	After	.21	.36	.46
Z.....	Before	.18	.10	.29
	After	.18	.38	.67

[1] Nueces Station is input station. Product filtered here was filtered ahead of point of injection of corrosion inhibitor.

TABLE 6—Analysis of Material Removed From Scraper Traps

Inhibitor	MATERIAL REMOVED FROM SCRAPER TRAPS					
	Percent Ash	Percent Iron	Percent Mill Scale	Percent Rust	Solids, Lbs	Rust, Lbs
W.....	92.6	59.2	82	18	3.5	0.63
X_1.....	100.0	70.0	77	23	5.25	1.21
X_2.....	101.5	70.8	80	20	5.63	1.13
Y.....	101.6	70.9	99	1	13.	0.14
Z.....	90.0	63.2	40	60	1.62	0.97

TABLE 7—Turbine Test Results

Inhibitor	Test Coupon Corrosion Rating	
	Nueces	Luling
W	A, B++, A	A, B+, B+
X_1	B+, B+	C(28%), B (9%)
X_2	A	B
Y	A, B++, A	A, B+, A
Z	A, B++, A	A, B+, A

TABLE 8—C Factors Obtained With Various Inhibitors

Inhibitor	C Factor
W	155.0
X_1	155.2
X_2	155.8
Y	156.7
Z	156.8

This caking was a phenomenon not exhibited by the corrosion product with the other inhibitors tested. The solids which gave rise to the rapid screen plugging characteristic were of a different consistency, being bulky and amorphous in structure. If not removed by use of strainers, this unusual type of corrosion product might cause difficulty in pipe or distribution systems elsewhere. The high frequency of plugging was not evident until about 25 days after the start of the test of inhibitor Z. The increased rate of corrosion, therefore, cannot be attributed to incompatibility of inhibitor Z with the previously tested inhibitor Y.

Modified Turbine Type Rust Tests

To evaluate the effectiveness of the corrosion inhibitor in quantities of products taken from the line at various points, a modification of the ASTM D-665 test, "Evaluation of Rusting Characteristics of Steam Turbine Oils in the Presence of Water," was used.[1, 2, 5] The test was modified by shortening it to 3½ hours and by reducing the temperature to 100 F.

This test is more severe in many respects than actual pipe line conditions. It is run with a much higher relative amount of water present than occurs in pipe line operations, and it is saturated with oxygen from the air at all times. The coupons from this test were visually rated, as were the in-line coupons, and were graded in the same fashion using the ratings of Table 2.

Numerous turbine tests were made during tests of each inhibitor, (see Table 7). Ratings of up to 5 percent corroded area, A, B++ and B+, are considered to be an indication that no significant amount of corrosion is occurring inside the pipe line. These data indicate that with the exception of inhibitor X_1, sufficient inhibitor was present in the products at the end of the pipe line to provide adequate protection. Inhibitor X_1 showed a significant depletion at the end of the line, but this possibly resulted from removal of considerable quantities of inhibitor from the pipe line by adsorption on the fine particles of sediment carried in the line during that portion of the test.

C Factor or Roughness Factor

An empirical formula developed by Hazen and Williams was used to evaluate the various inhibitors. This formula gives the surface roughness factor, or C factor, when the pressure drop due to friction, the API gravity at operating temperature, the rate of flow, and the inside pipe diameter are known or determined. (See Appendix for description.) A high factor indicates a smooth surface, while a low factor indicates a rough surface.

Table 8 shows C factors obtained during tests of each inhibitor.

These data indicate that the C factor gradually increased during the series of tests. C factors are known to be improved by scraper runs and also noticeably affected by slight errors in data taken for their calculation. The gradual increase in factor, therefore, can probably be attributed to repeated scraper runs at the end of each test. The data also indicate that none of the inhibitors permitted sufficient corrosion during the entire test period to cause a decrease in factor. Over a long period of time, the differences in corrosion protection afforded by the various inhibitors would probably cause a more pronounced change in factor.

Miscellaneous Observations

Laboratory tests showed that none of the inhibitors had an adverse effect on other properties of the products such as tetraethyl lead susceptibility, oxidation stability, or product color or odor.

No difficulty was encountered in handling characteristics of any of the inhibitors tested. All of the inhibitors tested were in a liquid form that remained uniform throughout the test period. Inhibitor Y was more viscous than the others, but no injection difficulties were encountered with it.

No difficulty of any kind that could be traced to incompatibility of the inhibitors was encountered during the entire test program.

All the products received from the refinery must pass through 60 mesh strainers before they are metered at the point where they are received by the pipe line system at Nueces Station. Prior to these tests, all products were inhibited during blending at the refinery, thereby providing corrosion protection to refinery products storage tanks. At the end of the test program, the incoming screens at Nueces Station were beginning to plug from rust scale formed in refinery tankage and headers during the handling of uninhibited products. The frequency of cleaning increased from two to three times a year to three times a month at the conclusion of this series of tests.

The effects of a change in inhibitors did not become apparent in any of the tests until the new inhibitor had been injected in all products entering the pipe line for at least a two week period. This indicates that some inhibitor remains in the system and some corrosion protection is provided by it for a length of time following its use. Data obtained during the first two weeks after a change in inhibitors were therefore not used for evaluation purposes.

Conclusions

The results of the tests indicated that inhibitor Y was superior to the other inhibitors, since it provided protection during both static and dynamic conditions. Inhibitor W was next in rating by each method, and inhibitor Z provided a high level of protection but allowed formation of a small amount of bulky, amorphous corrosion product which caused frequent strainer plugging.

Of the seven methods of evaluation used, the in-line test coupon method proved to be the most satisfactory and practical. The frequency of strainer plugging and measurement of scraper trap deposits are relative methods only, because they are subject to changes in operating conditions. The C factor method is not satisfactory for a short term test but will indicate general overall level of protection.

Analysis by X-ray diffraction methods of the solids removed by filtration provided data on the relative amounts of corrosion products formed in the pipe line during the tests.

Acknowledgment

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Appendix 1—Calculation of C Factor

$$P^{0.54} = \frac{162.04\ Q\ g^{0.54}}{C\ d^{2.63}}$$

where:

P = Pressure drop resulting from friction only in pounds per square inch gauge per mile of pipe.
Q = Rate of flow in barrels per hour
g = Specific gravity of the product at line temperature
d = Inside diameter of pipe in inches
C = C Factor

Any discussions of this article not published above will appear in the June, 1959 issue

Factors Influencing the Rate-of-Pickling Test on Tin-Plate Steel*

By R. M. HUDSON and G. L. STRAGAND

Abstract

"Lag time" is a measure of the time of pickling necessary to produce a constant rate of weight loss from steel immersed in acid. This measurement has been used as a guide for improving the corrosion resistance of commercial electrolytic tin plate. It is determined by measuring either the rate of change of weight loss, hydrogen evolution, or corroding potential of a specimen in 6N hydrochloric acid at 90 C (194 F.)

The lag time depends on surface effects inasmuch as removing the surface layers of steel by abrasion or by pickling destroys the lag. The influence of box-annealing atmospheres, cleanliness of steel, and time-temperature cycles on lag time have been investigated, and the complexity of these effects has been demonstrated. Explanations in terms of oxidation or decarburization of the steel surface during annealing are not feasible for the development of lag time under all the experimental conditions studied.

Preliminary data demonstrating the high concentrations of certain elements on the steel surface before annealing, and the enrichment of the surface layer by some of these elements during annealing, are suggested as particularly promising areas for future work. In this way lag time phenomena in tin-plate steels may be better understood and further improvement in tin-plate corrosion resistance can be made. 2.3.4

Introduction

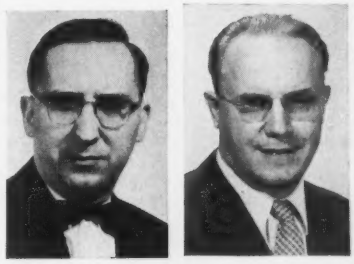
TIN PLATE represents one of the major items produced by the steel industry in the United States. Tin plate is thin (0.01-inch) low-carbon steel covered on each side by a much thinner 0.000015- to 0.00009-inch) layer of pure in, with strata of iron-tin alloy between. The consumption of tin plate has increased steadily from about 340,000 tons n 1900 to more than 5.5 million net tons n 1955. This increase has kept pace with he growth of the food-packaging industry because the largest use of tin plate is or food containers. Although all tin plate formerly was made by the hot-dip process, in recent years about three fourths of the total production has been electrolytic tin plate, which can be produced with lighter tin coatings than the hot-dip product.

The successful application of tin plate for containers depends to a large extent on its corrosion resistance. Food products react with the tin coating and the base metal, but the rate of reaction generally is sufficiently slow for the service life of a container to be longer than the time required for distribution and consumption. Service life may be defined as the time required for corrosion to liberate sufficient hydrogen to bulge the can ends or to perforate the container. The corrosion of tin-plate containers by food products is a complex problem, and much of the knowledge of the problem is still empirical. Among the major factors that affect the rate at which tin plate corrodes are the type of food product, the packing procedure used in canning, and the storage conditions. In a mildly acidic, oxygen-free environment, such as is present in most canned foods, tin is anodic to iron and normally provides electrochemical protection to the small exposed areas of steel.[1,2,3] Differences between good and poor corrosion resistance in plain cans may be interpreted therefore as differences in the ability of the steel to receive galvanic protection.

The rate-of-pickling test (designated as lag-time test in the present paper) was developed by the American Can Company[4] as a guide to evaluating the corrosion resistance of commercial electrolytic tin plate. In this test a specimen (detinned or untinned) is immersed in 6N hydrochloric acid at 90 C (194 F), and the time (lag time) required for a uniform rate of attack to develop is estimated. One method of testing involves weighing, pickling for definite time intervals, and reweighing. For convenience, an instrument known as the "Autographic Pickling Rate Tester," which draws the rate of pickling curve automatically, was developed by the American Can Company. This instrument is activated by the hydrogen pressure developed from a specimen dropped into a closed vessel of acid. Koehler[5] has described a rapid test for lag time, in which the corroding potential of the steel specimen in the hot hydrochloric acid is measured with respect to a reference electrode. Steels with a low lag are attacked at a uniform rate early in the test, whereas steels with a higher lag are initially attacked at a rate considerably slower than the final linear rate. Typical curves for steels with high and low lags are shown in Figure 1.

The lag time has been reported to depend upon the annealing conditions. Evidence has been presented[4,5] which demonstrates that tin plate having good performance in pack tests (made to determine the actual performance of containers packed with a food product, such as prunes or peaches) usually has a low lag time. The lag time test is not represented as providing a total evaluation of tin-plate corrosion resistance, but as measuring one specific, contributing factor.

The purpose of this paper is to indicate the factors, cleaning and annealing in particular, that influence the lag time of tin-plate steel, and to describe mechanism studies that were made to aid in understanding the phenomenon. It should be emphasized that all these studies were made in the laboratory; therefore, the cleaning and annealing procedures, although made under careful control, may not closely simulate commercial operations. For example, the exposure of specimens to the gas atmospheres used during laboratory annealing cannot be expected to duplicate the exposure of a tightly wrapped coil to the atmosphere during commercial annealing.



Hudson Stragand

R. M. HUDSON — Section Head, Physical Chemistry Section, Chemical-Metallurgy Division Applied Research Laboratory, United States Steel Corp., Monroeville, Pa. He has a BS in chemistry, 1947, and a PhD in chemistry from Yale in 1950. His technical experience includes work in electrochemistry, gas-metal reactions during heat treatment of steel, corrosion studies on tin plate and hydrogen beheavier than steel.

G. L. STRAGAND—Assistant Technologist, Applied Research Laboratory, United States Steel Corp., Monroeville, Pa. He has a BS in chemistry, University of Pittsburgh, 1943 and an MS in chemistry from the same school, 1950. His experience includes analyses of steels, alloys and raw materials at Clairton Works, U. S. Steel, teaching of microchemistry at the University of Pittsburgh, and his present work on gas-metal reactions during annealing of steel, tin-plate corrosion, and studies on hydrogen in steel.

Materials and Procedures

Specimens used for laboratory cleaning-and-annealing experiments were cut from cold-reduced tin-plate steel and specimens for other laboratory experiments were cut from commercially box-annealed tin-plate steel. Several coil lots were used, to ensure that the observed effects would be general. All steels were of nominally the same grade; analyses are given in Table 1 for as-cold-reduced material used for laboratory cleaning-and-annealing experiments, and in Table 2 for the commercially box-annealed ma-

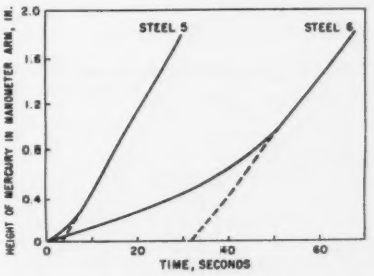


Figure 1—Typical lag-time curves showing high and low lag-time values.

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TABLE 1—Composition of Cold-Reduced Tin-Plate Steels Used

Sample No.	COMPOSITION, PERCENT							
	C	Mn	P	S	Si	Cu	Ni	Cr
A	0.110	0.44	0.004	0.033	0.008	0.03	0.02	0.01
B	0.095	0.55	0.006	0.034	0.004	0.06	0.03	0.03
C	0.140	0.49	0.009	0.023	0.006	0.03	0.02	0.01
D	0.108	0.49	0.009	0.028	0.005	0.03	0.02	0.02
E	0.097	0.40	0.011	0.045	0.005	0.05	0.05	0.03
F	0.085	0.48	0.009	0.036	0.003	0.04	0.03	0.02
G	0.115	0.55	0.009	0.025	0.005	0.03	0.02	0.01
H	0.095	0.39	0.006	0.030	0.004	0.03	0.02	0.01
I	0.100	0.50	0.012	0.024	0.004	0.01	0.01	0.01
J	0.086	0.36	0.014	0.051	0.004	0.01	0.01	0.01
K	0.068	0.43	0.013	0.041	0.004	0.05	0.04	0.02
L	0.054	0.38	0.011	0.045	0.004	0.03	0.03	0.02
M	0.050	0.38	0.009	0.023	0.004	0.02	0.02	0.02
N	0.063	0.33	0.014	0.035	0.004	0.01	0.01	0.01
O	0.110	0.47	0.011	0.039	0.009	0.03	0.01	0.01
P	0.120	0.53	0.006	0.033	0.003	0.02	0.01	0.02
Q	0.080	0.36	0.006	0.025	0.006	0.02	0.02	0.01
R	0.088	0.41	0.008	0.037	0.010	0.02	0.01	0.02
S	0.110	0.50	0.011	0.018	0.020	0.02	0.00	0.04
T	0.070	0.33	0.011	0.028	0.010	0.07	0.05	0.04
U	0.080	0.38	0.016	0.030	0.006	0.03	0.00	0.02
V	0.060	0.33	0.008	0.027	0.010	0.06	0.08	0.01

terial. Condition of the steel used in laboratory annealing experiments included: (1) Uncleaned steel (cold-reduction lubricants present), (2) Solvent-cleaned steel (carbon tetrachloride), (3) Vapor-degreased steel (trichloroethylene), (4) Electrolytic-alkaline cleaned (commercial silicate and phosphate cleaners) and water-rinsed steel, and (5) Electrolytic-alkaline cleaned steel with dried-on cleaner. Laboratory annealing experiments in atmosphere-controlled furnaces were of two types:

1. Short heating (111 C/hr or 200 F/hr) and cooling (56 C/hr or 100 F/hr) cycles. Enough fresh gas was supplied by barostatic control to maintain 1 atmosphere pressure in the furnace. (In some instances, the furnace gas was recycled by a gas pump.)

2. Long heating (19 C/hr or 35 F/hr) and cooling (28 C/hr or 50 F/hr cycles. A continuous flow (1 cubic foot per hour) of fresh gas was supplied at 1 atmosphere pressure. Moisture content of the gas mixtures used was controlled by passage through temperature-controlled salt solutions or a magnesium perchlorate drying train. In the annealing tests, both coupons (8 x 65 mm) and panels (15 x 33 cm) of steel were used in stacks of 30 to 40 which were centered in controlled-atmosphere furnaces. Usually material from several lots of steel was annealed simultaneously.

The effect on lag time of oxidizing steel by heating in air at temperatures from 204 to 482 C (400 to 900 F) was studied by using an oven with air-circulating controls. By means of pickling, surface analyses were determined on layers of steel removed from several lots of unannealed, commercially box-annealed, and laboratory-annealed steels. The effect on lag time of removing surface layers of steel by pickling prior to laboratory annealing was studied in addition to the effect on lag time of removing surface layers from specimens of annealed steel prior to the determination of lag time.

Hydrogen is known to enter steel during acid-pickling,[6] and therefore the relationship to lag time of hydrogen content in the steel during immersion of specimens in 6N HCl at 90 C (194 F) was examined. Separate specimens were immersed for various times in acid after which they were removed, rinsed with distilled water and either placed in borosilicate-glass hydrogen-extraction burettes in which they were heated for 16 hours at 160 C (320 F) under mercury or given the lag-time test. The hydrogen extracted from the steel was collected over mercury and measured volumetrically after which the specimens were weighed and the hydrogen content expressed in cubic centimeters (STP) per 100 grams of steel (1 cm^3 (STP)/100 g is equivalent to 0.9 parts per million.) This[7] extraction method is similar to that of Darken and Smith.[8]

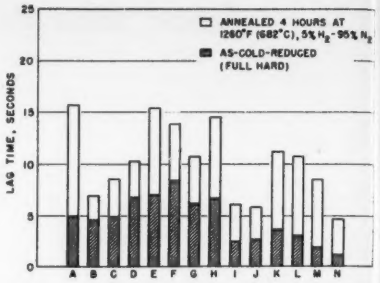

Figure 2—Lag time of annealed and of as-cold-reduced tin-plate steel.

Results and Discussion

Development of Lag Time During Annealing

Willey, Krickl, and Hartwell[4] have shown that lag time arises in the box-annealing operation and that it is present in the surface layers of metal to varying depths, but generally to not more than 30 or 40 millionths of an inch. A group of as-cold-reduced steels were given a combination of electrolytic cleaning and solvent cleaning (with carbon tetrachloride), and specimens were tested for lag time before and after a laboratory annealing treatment at 682 C (1260 F) for 4 hours using the short heating and cooling cycle and a gas mixture of 5 percent hydrogen containing nitrogen (less than 0.04 percent water). The results of these tests are presented in Figure 2.

The cold-reduced steel had an initial lag time and, although this lag time was usually low, one steel had a lag as high as 8 seconds. In all cases, annealing increased the lag time, but the response of all steels was not the same, even though the annealing operations were identical with respect to temperature, gas atmosphere, and geometry of the steel coupons within the furnace. The differences observed among annealed steels and among the unannealed specimens indicate that annealing conditions and such factors as chemical composition and processing of the steel through the cold-reduction stage influence the lag time.

Chemical Composition and Lag Time

Surface layers were stripped from four lots of commercially box-annealed tin-plate steels and analyses made on the resultant solutions so that surface-layer composition could be compared with the bulk composition of the steel. The amount of steel stripped from these ma-

TABLE 2—Composition of Commercially Box-Annealed Tin-Plate Steels Used

Sample No.	COMPOSITION, PERCENT					
	C	Mn	P	S	Si	Cu
1	0.071	0.38	0.009	0.036	0.001	0.01
2	0.078	0.38	0.010	0.036	0.001	0.01
3	0.058	0.28	0.005	0.028	0.001	0.05
4	0.074	0.30	0.005	0.036	0.001	0.04
5	0.088	0.49	0.006	0.023	0.001	0.06
6	0.100	0.34	0.006	0.040	0.001	0.07
7	0.075	0.45	0.008	0.034	0.002	0.033
8	0.074	0.43	0.010	0.025	0.001	0.002

TABLE 3—Comparison of Surface and Bulk Compositions of Commercially Box-Annealed Tin-Plate Steel

Sample No.*	Source	COMPOSITION, PERCENT						
		Si	Cu	Mn	Al	Ni	Cr	Mo
1	Surface	0.27	0.010	0.97	0.037	0.010	0.014	0.002
	Bulk	0.001	0.005	0.36	0.029	0.007	0.011	0.001
4	Surface	0.23	0.043	0.58	0.066	0.049	0.024	0.004
	Bulk	0.001	0.022	0.30	0.021	0.038	0.017	0.003
7	Surface	0.14	0.060	0.65	0.047	0.039	0.024	0.005
	Bulk	0.002	0.033	0.45	0.024	0.034	0.019	0.003
8	Surface	0.18	0.007	0.53	0.035	0.010	0.010	0.001
	Bulk	0.001	0.002	0.43	0.027	0.008	0.008	0.0005

* Listed in Table 2.

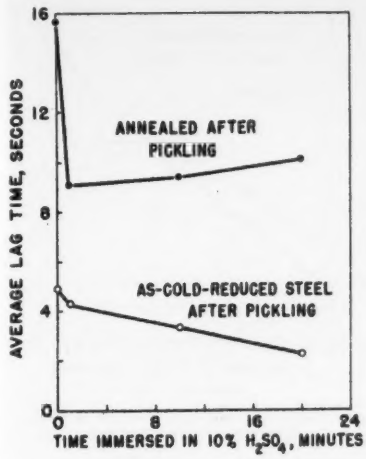

Figure 3—Effect of pickling prior to annealing on resultant lag time.

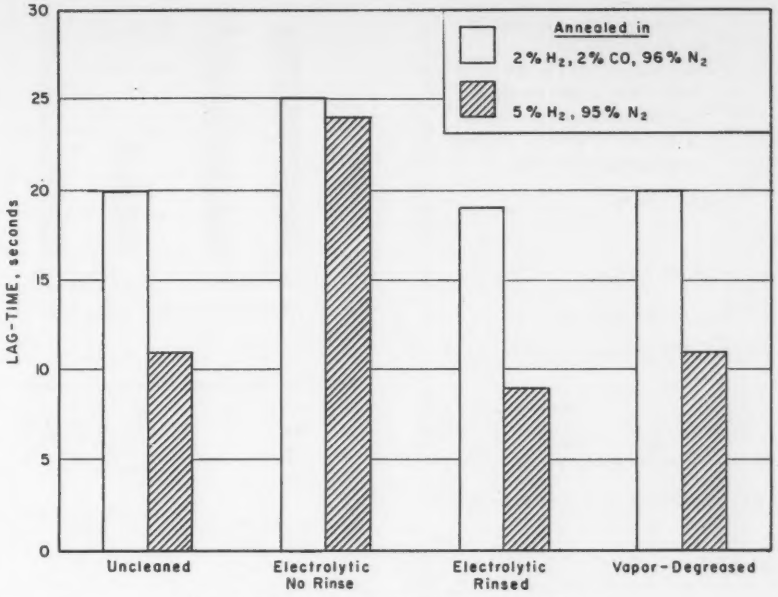

Figure 4—Effect on lag time of cleaning before annealing.

terials was 0.3, 0.8, 0.4, and 0.4 percent by weight respectively. The results in Table 3 clearly show that the surface layer of these steels contains a high concentration of silicon as compared to that of the base steel, and a significantly higher concentration of copper, manganese, and aluminum. Koehler[5] has also reported that the surface layer of tinplate steel is rich in copper, manganese, aluminum, silicon, and nickel.

The results of surface analyses for copper, sulfur, and phosphorus on two lots of laboratory annealed steels are shown in Table 4 and these results are compared with those for the corresponding cold-reduced material. Approximately 0.5 percent by weight of each sample from one surface only was removed by pickling. Bulk analyses were made on the total steel cross section by chemical methods. The surface concentration of copper was greater than the bulk concentration for both unannealed and annealed specimens; annealing tended to concentrate copper on the surface. Surface and bulk concentrations of phosphorus for unannealed specimens were nearly identical, but annealing tended to concentrate phosphorus on the surface. For some chemical constituents, therefore, the few specimens examined showed a real difference between surface and bulk concentration in both the as-cold-reduced and the annealed conditions. An explanation for the enrichment of the surface layer by certain constituents during annealing is not presently known.

Although significant differences in surface composition were observed between steel lots and between the surface and bulk compositions of each steel lot, no correlation between composition and lag time was found.

Effect on Lag Time of Pickling Steel Prior to Annealing

A chemical pickle prior to box annealing of the steel was found to be effective in lowering the lag time of annealed samples. For this study, electrolytic- and solvent-cleaned specimens were pickled and then rinsed with water and dried prior to annealing for 4 hours at 682 C (1260 F) using the short heating and cooling cycle and a gas mixture of 2 percent hydrogen, 2 percent carbon monoxide, and 96 percent nitrogen. Pickling was done in 10 percent H_2SO_4 at 70 F for 1, 10 and 20 minutes. This removed 0.02, 0.2, and 0.4 percent of the steel representing 2, 20, and 40 millionths of an inch of steel. The resultant lag times are shown in Figure 3.

It is evident that removing the initial surface layer of the steel by pickling prior to annealing influences the magnitude of the lag developed during the annealing operation. When the initial steel surface was not removed before annealing, a substantially higher lag time resulted. This experiment is further evidence for the existence on the steel of a surface layer that influences lag time.

TABLE 4—Comparison of Surface and Bulk Compositions of Tin-Plate Steel

Steel	Treatment	SURFACE ANALYSIS, PERCENT			BULK ANALYSIS, PERCENT		
		Cu	P	S	Cu	P	S
H	Unannealed	0.058	0.0060	0.033	0.034	0.006	0.030
H	Annealed 4 hours 682C (1260F) in 5% H_2-95% N_2	0.113	0.0113	0.040	0.032	0.003	0.033
M	Unannealed	0.066	0.0114	0.030	0.017	0.009	0.023
M	Annealed 4 hours 682C (1260F) in 5% H_2-95% N_2	0.095	0.0179	0.037	0.017	0.008	0.026

TABLE 5—Effect on Lag Time of Heating Tin-Plate Steel in Air (1 ft³/hr; less than 0.2% water)

Steel	Baking Temperature		Lag Time (seconds) for Each Bake Time (minutes)							
	C	F	½	1	5	10	30	60	120	240
"Low Lag"	204	400	5	..	..	4	..	4	5	6
	288	550	..	5	4	5	3	3	..	..
	316	600	5	4	4	4	4	4	..	..
	399	750	5	3	3	4	4	..	..	..
	482	900	5	4	3	3	6	..	..	..
"High Lag"	204	400	34	31	31	31	33	31	29	30
	288	550	30	30	29	38	29	..	..	..
	316	600	29	30	26	29	28	..	..	..
	399	750	29	30	30	31	..	..	..	..
	482	900	24	27	13	12	..	..	..	..

Effect of Air-Formed Oxide Films on Lag Time

Of the various gas-metal reactions that may occur during an annealing treatment, one is oxidation of the steel by either water vapor, or carbon dioxide, or free oxygen. To learn whether the amount of oxide formed on steel affects lag time, solvent-cleaned specimens of steel, representing relatively "low" (4 seconds) and "high" (32 seconds) lag times respectively, were heated in air for various times at temperatures in the range 204 to 482 C (400 to 900 F).

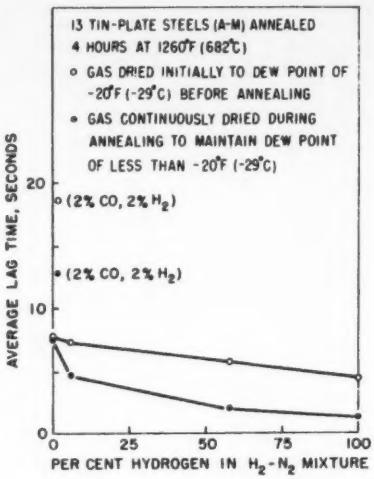
13 TIN-PLATE STEELS (A-M) ANNEALED
4 HOURS AT 1260°F (682°C)
o GAS DRIED INITIALLY TO DEW POINT OF -20°F (-29°C) BEFORE ANNEALING
• GAS CONTINUOUSLY DRIED DURING ANNEALING TO MAINTAIN DEW POINT OF LESS THAN -20°F (-29°C)
o (2% CO, 2% H2)
• (2% CO, 2% H2)
AVERAGE LAG TIME, SECONDS
PER CENT HYDROGEN IN H2-N2 MIXTURE

Figure 5—Effect of gas atmosphere on lag time.

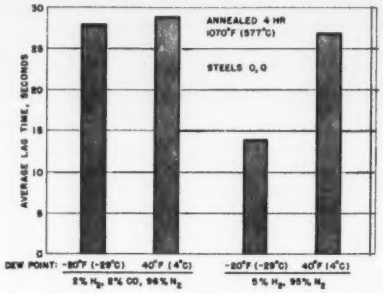

Figure 6—Influence of moisture in gas on lag time of tin-plate steels.

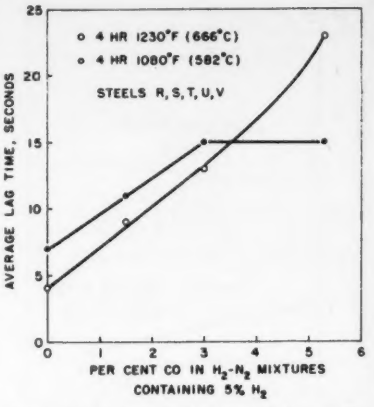

Figure 7—Influence of carbon monoxide in annealing atmospheres on lag-time effect.

TABLE 6—Maximum Moisture Content

Annealing Atmosphere	Maximum Moisture Content During Laboratory Annealing Tests, Percent	
	Pre-Dried Gas	Continuously-Dried Gas
100% N_2	0.07	0.01
2% H_2, 2% CO, 96% N_2	0.8	0.03
6% H_2, 94% N_2	0.5	0.02
58% H_2, 42% N_2	0.9	0.01
100% H_2	0.9	0.02

After these treatments, lag times were determined on the oxidized specimens. The results of these tests are given in Table 5.

It is noteworthy that even though oxide films of a blue or gray color were formed on specimens heated at temperatures in the range 204 to 399 C (400 to 750 F) the lag times of these specimens were similar to those of specimens that were not heated. An air-formed oxide film in itself does not appear to be a factor in regard to lag time when the oxide is formed in this temperature range. Heating specimens having an initially high lag time at 482 C (900 F) resulted in a significant lowering in lag time, even though these specimens were covered with a gray oxide scale. This scale was easily removed by the hot hydrochloric acid, probably because of acid attack through discontinuities in the scale. A similar observation has been reported by Koehler.[5] One explanation for this lowering in lag time is that the oxide scale consisted of a considerable amount of the original surface layer; therefore, because the oxide scale was rapidly undermined and removed, the effective thickness of the remainder of the surface layer would be less, and a lower lag time would be obtained. Oxides formed by other reactions (such as water vapor reacting with iron) may, however, not be of the same form and may have a different influence on lag time.

Effect of Cleaning Prior to Annealing on Lag Time

Laboratory cleaning experiments were conducted on steel O and comparisons were made of the lag time after annealing of (1) specimens that had received no cleaning (cold-reduction lubricants present), (2) specimens that were electrolytically cleaned, (3) specimens that were electrolytically cleaned but not water-rinsed to remove cleaner residues (these residues were dried on the panels), and (4) specimens that were vapor degreased. Annealing of these specimens was conducted for 4 hours at 554, 577, and 632 C (1030, 1070, and 1170 F) using the long heating and cooling cycle and gas mixtures containing 2 percent hydrogen, 2 percent carbon monoxide, and 96 percent nitrogen (less than 0.1 percent water), and in gas mixtures containing 5 percent hydrogen and 95 percent nitrogen (less than 0.1 percent water). The results shown in Figure 4 have been averaged over the three annealing temperatures.

The highest lag times were obtained for specimens that were electrolytically cleaned without rinsing before being annealed. The difference in lag time between uncleaned and cleaned lots was small. Lower lag times were obtained for specimens that were annealed in the hydrogen-nitrogen type atmosphere, except for the unrinsed electrolytic-cleaned specimens, which had high values regardless of the atmosphere, an indication that dried residues from the alkaline cleaning solutions "masked" the steel surface from the gas. The cleaning residues remaining on the surface were deliberately made heavy, and these residues probably became vitrified during the annealing operation. Only trace amounts of such materials would be expected on electrolytically cleaned and rinsed steel.

Effect of Annealing Cycle and Gas Atmosphere on Lag Time

Willey, Krickl and Hartwell[4] have shown that to remove the lag time of a steel without removing the surface layer of metal by acid dissolution, or by abrading and polishing the steel to a sufficient depth, the surface layer of the metal must be exposed to suitable atmospheres during heat treatment, dry hydrogen being particularly effective. They have shown that high lag times can be produced by annealing in wet hydrogen (30 percent water) and that subsequent reannealing in dry hydrogen can remove the lag time so produced.

The influence of moisture content in the annealing atmosphere on lag time is further indicated in Figure 5 for steels that were laboratory-annealed for 4 hours at 682 C (1260 F) using the short heating and cooling cycle. The atmospheres used are listed in Table 6. Lower lag times were obtained when the gas was continuously dried by recirculation through a drying train than when the gas was only pre-dried initially, the moisture level in the furnace gas being higher during the latter tests as shown in Table 6. The rise and subsequent fall of moisture content in the noncirculated atmosphere may be due to desorption of water from the furnace tube and the reaction of hydrogen with oxides on the steel. For the circulated atmosphere, moisture from these sources did not build up. Increasing the hydrogen content in the mixture caused a lowering in lag time. In Figure 6 results are shown for steels that were laboratory-annealed (long heating and cooling cycle) for 4 hours at 577 C (1070 F) using an atmosphere of 2 percent hydrogen, 2 percent carbon monoxide, and 96 percent nitrogen, and an atmosphere of 5 percent hydrogen and 95 percent nitrogen; the moisture levels studied were 0.04 and 0.8 percent water corresponding to dew points of —20 and + 40 F. Oxidation of the steel surface by moisture cannot be the only factor responsible for the development of lag time in steel. For the two gas mixtures used, high lag times were obtained when the moisture level was high, but when the moisture level was low, the lag time of specimens annealed in an atmosphere of 5 percent hydrogen and 95 percent nitrogen was considerably lower than the lag time of specimens annealed in an atmosphere of 2 percent hydrogen, 2 percent carbon monoxide, and 96 percent nitrogen.

Koehler[5] has reported that some annealing atmospheres which are not oxidizing to ferrite grains are oxidizing to grain boundaries, and that the determination of the lag time is, in effect, a test for the presence of grain-boundary oxides. His paper contains photomicrographs of this condition and an explanation of the mechanism of the lag time effect for such cases.

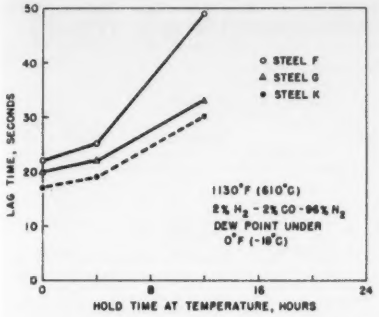

Figure 8—Effect of hold time on lag time of tin-plate steels (1130 F).

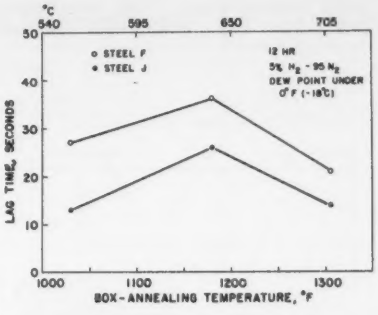

Figure 10—Effect of annealing temperature on lag time of tin-plate steels (5 percent H_2—95 percent N_2).

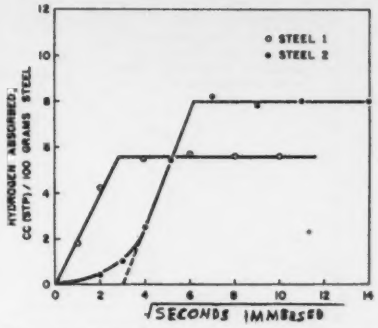

Figure 12—Typical hydrogen absorption rate curves in 6 N HCl at 90 C (194 F).

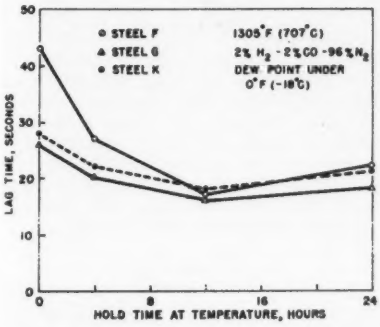

Figure 9—Effect of hold time on lag time of tin-plate steels (1305 F).

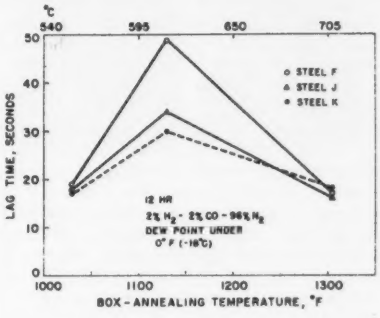

Figure 11—Effect of annealing temperature on lag time of tin-plate steels (2 percent H_2—2 percent CO—96 percent N_2).

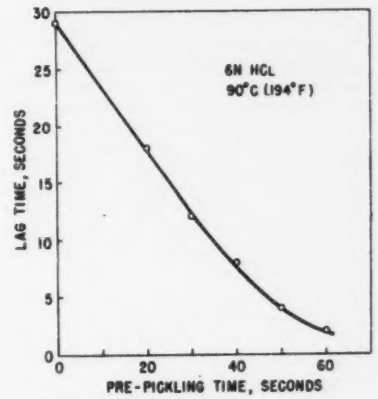

Figure 13—Effect of pickling a specimen on the lag time.

The presence of carbon monoxide in annealing atmospheres, even though moisture content is low, has been shown repeatedly to result in relatively high lag time values compared with the values obtained by using carbon monoxide-free, dried hydrogen-nitrogen atmospheres. The results of laboratory annealing experiments (long heating and cooling cycle) in which the amount of carbon monoxide present in hydrogen-nitrogen mixtures containing 5 percent hydrogen and less than 0.1 percent water was studied, are shown in Figure 7. The lag time of specimens annealed in atmospheres containing little or no carbon monoxide is lower than the lag time of specimens annealed in atmospheres containing carbon monoxide. The influence of annealing temperature is also indicated here. Time-temperature effects on lag time will be discussed later.

The results shown in Figure 7 are difficult to explain in terms of surface oxidation. As the carbon monoxide content of these atmospheres was increased (the hydrogen content being held constant), the atmosphere remained reducing to iron over the temperature range studied, yet higher lag times resulted. This is in contrast to the observed decrease in lag time when the hydrogen content in the atmosphere is increased.

The effect of holding time and holding temperature on lag time is shown in Figures 8 through 11 for laboratory annealed specimens (long heating and cooling cycle). Obviously, the relationship of annealing time and temperature to lag time is not simple. If the development of the lag-time effect were only a function of decarburization or oxidation reactions at the steel surface, steady increase or decrease in lag time might be expected with increasing hold time and increasing temperature, the effect being dependent on the particular atmosphere used. The presence of maxima or minima in lag time as a function of hold time or temperature may be due to diffusion of certain elements, such as copper and phosphorus, to the surface of the steel. The change in lag time from an increasing to a decreasing rate, or vice versa, may be related to subsequent reaction of nonmetallic elements with the gas atmosphere at or near the steel surface. The ability of steel to evolve gases during annealing is well known, and the rate of evolution of gas has been observed by the authors to increase and subsequently to decrease during commercial box-annealing cycles.

Hydrogen Absorption by Steel During Pickling

During acid pickling it is well known that steels will absorb hydrogen, the amount depending on the pickling conditions and on the prior processing of the steel.[6] To study the relationship between hydrogen absorption in the steel and lag time, hydrogen-absorption rates were measured in 6N hydrochloric acid at 90 C (194 F) for six commercially box-annealed steels having known lag times.

In Figure 12, hydrogen absorption rate curves (hydrogen concentration in the steel plotted against the square root of the time the specimen had been immersed in acid) in 6N hydrochloric acid at 90 C (194 F) are shown for two of the steels studied. For some of the steels there was an induction period before a uniform rate was established. In Table 7, lag time values (time intercepts) from hydrogen evolution rate curves (Figure 1) and the inflection points in these curves are compared with the time intercepts and inflection points from the corresponding hydrogen absorption rate curves and with the time required to saturate the steel with hydrogen.

The inflection points in the hydrogen absorption-rate curves are similar to the inflection points in the hydrogen evolution rate curves. The intercepts from the hydrogen absorption-rate curves are similar to the lag times determined from the intercepts of the hydrogen evolution-rate curves, but are generally lower. For all these steels, the time required to saturate a steel with hydrogen was considerably longer than the lag time; this indicates that lag time is not an induction period corresponding to the time required for the steel to become saturated with hydrogen.

Effect of Earlier Pickling on Lag Time and Hydrogen-Absorption Rate

As shown in Figure 13, pickling a specimen prior to testing reduced its lag time as has been reported previously.[4,5] Figure 14 shows that no induction period

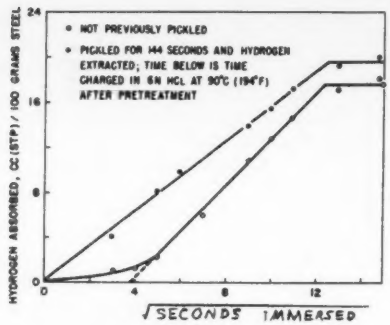

Figure 14—Hydrogen absorption rate curves for steel specimens with and without prepickling to remove initial surface.

before establishment of a uniform hydrogen absorption rate was observed for specimens from which the surface layer was initially removed by pickling. (After the hydrogen initially introduced into these specimens during pickling was extracted by heating under mercury, the specimens were immersed in 6N hydrochloric acid at 90 C (194 F) for various times, after which the hydrogen content was determined.) This may be due to differences in chemical composition between the surface layer and the remainder of the steel cross section or to a change in steel surface reactivity due to pickling. The same characteristic of the steel sample that produces the lag-time effect in establishing uniform hydrogen-evolution rates and weight-loss rates is probably responsible for the induction period preceding uniform hydrogen absorption rates. This characteristic may best be described as a surface effect that does not persist after the original steel surface has been removed.

Conclusions

A study has been made of the factors, cleaning and annealing in particular, that influence the lag time of tin-plate steel.

TABLE 7—Comparison of Lag Time Value with Hydrogen-Absorption-Rate Characteristics of Specimens Immersed in 6N HCl at 90 C (194 F)

Steel	INDUCTION TIME, SECONDS				Time to Saturate Steel with Hydrogen, sec
	From Hydrogen Evolution Rate		From Hydrogen Absorption Rate		
	Intercept	Inflection Point	Intercept	Inflection Point	
1	0	1	0	0	8
2	10	14	9	14	38
3	4	7	2	4	12
4	21	36	14	27	61
5	3	5	1	4	10
6	32	50	23	49	135

Mechanism studies have been made to aid in understanding the phenomenon.

Cold-reduced steel has a low initial lag time that is usually increased by annealing. The response of all steels was not the same even though annealing operations were identical. Cleaning before annealing had little effect on lag time although the presence on the steel surface of dried residues from alkaline cleaning solutions did result in higher lag times for the annealed steel. The influence of annealing atmosphere composition was important, lower lag times resulting when the moisture and carbon monoxide contents in the protective atmosphere were low and the hydrogen content in the atmosphere was high. An air-formed oxide film in itself did not appear to be a factor in regard to lag time when the oxide was formed in the range 204 to 399 C. The influence of annealing time and temperature on lag time has been shown to be complex, and explanations of the development of lag time in terms of oxidation or decarburization of the steel surface during annealing seem unlikely to apply to all instances reported.

It is concluded that lag time depends on surface effects that may be partially explained in terms of a uniformly affected surface layer. Removal of the steel surface, such as by pickling, was found to result in lower lag times. Lag time is not an induction period corresponding to the time required to saturate the steel with hydrogen during acid immersion. The higher initial concentration of certain elements found in the surface layer of commercial steels and the enrichment of the surface layer of steel in both copper and phosphorus by laboratory annealing has been reported. Improved microchemical analyses of surface concentrations of these and other elements may help in achieving a better understanding of the origin of lag time in tin-plate steels and may also result in improvements in tin-plate corrosion resistance.

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High Temperature Oxidation of Chromium-Nickel Steels*

By D. CAPLAN and M. COHEN

Abstract

The scaling of austenitic stainless steels Type 302, 309 and 330 has been investigated by weight gain vs time measurements in air at 1600 to 2000 F and subsequent examination of the scales. As had been found previously with chromium steel, the curves exhibit breaks indicating intermediate periods of rapid oxidation due to disruption of the protective scale layer. Accumulation of silica at the metal/scale interface is found to contribute to this disruption; voids are considered to have the same effect. A distinction is drawn between such breaks and the type which arises from the extraordinary protectiveness of an initial thin oxide film, which is markedly affected by surface preparation and prior treatment.

3.2.3

Introduction

AN EARLIER publication[1] reported on the scaling characteristics of three chromium steels. The principal finding was that breaks in the weight gain/time curves could be related to the accumulation of silica as a separate phase at the metal/scale interface; periods of accelerated oxidation resulted from the cracking of the protective scale after a silica layer had isolated it from the metal specimen. The present work is an extension of these experiments to the chromium-nickel steels. In the scaling experiments, as before, the weight increase of the specimens was followed continuously with time. The scales were examined microscopically and by X-ray diffraction and analyzed colorimetrically and spectrographically.

Experimental

The chemical composition of the alloys used is shown in Table 1.

Details of specimen preparation and experimental procedure have been described.[1] Cylindrical specimens ¼ inch by 1¼ inch long were machined from rod and abraded through No. 00 paper. Most were then electropolished in perchloric-acetic electrolyte; specimens 8, 19, 20 and 21 were simply scrubbed and degreased after being abraded.

The scaling runs were carried out in a vertical tube furnace with the specimens suspended from an analytical balance. Weighings were made at suitable intervals. A prepared atmosphere of dry air or air saturated with moisture at 90 F flowed upward through the tube at 200 ml/min, equivalent to a linear rate of 12 inches/min at room temperature or 56 inches/min at 2000 F.

Most runs were begun by lowering the specimens into the hot furance with the prepared atmosphere flowing; five to ten minutes were then required before the apparatus returned to the test temperature. Specimens 8, 20 and 21 were brought to temperature by heating in flowing helium for 30 minutes. Zero time was when the helium was turned off and air turned on.

The specimens were observed during the run by means of a 45 degree mirror and telescope at the open end of the furnace tube; local oxide eruptions in the form of spines or warts could be detected in this way. Test temperatures were 1600, 1800 and 2000 F. Specific information for the individual runs is included with the table of results, Table 2.

Cohen

Caplan

MORRIS COHEN—After experience as a chemist at a battery manufacturing plant, he joined National Research Council of Canada, Ottawa. He has been head of the Corrosion Section, Division of Applied Chemistry since 1946. He has a PhD from University of Toronto, 1939 and has authored numerous articles on corrosion in aqueous solutions and on high temperature oxidation.

DONALD CAPLAN is with the corrosion laboratory of the National Research Council of Canada, Ottawa, where his principal interests have been corrosion of stainless steels, scaling of metals and corrosion by anti-freeze solutions. He has a BSc and MSc from Queen's University, Kingston and a PhD in metallurgy, Rensselaer Polytechnic Institute, 1956.

A stereomicroscope was used to observe the scaled specimens as they cooled after removal from the furnace. Subsequently, the various scale layers were sampled, as independently as possible, for X-ray diffraction and spectrographic analysis. Some colorimetric chemical analyses were made to assist the interpretation of the micro-spectrographic results. Metallographic sections of the scaled specimens were prepared by a method previously described[2] and photomicrographs taken of typical areas.

Results

Figures 1 to 3 show the scaling rate curves of Type 302, 330 and 309 steels. To minimize confusion many of the experimental points have not been plotted. The similarity of the curves for the seven sets of duplicates (3-4, 6-7, 9-10, 11-12, 14-15, 16-17, and 20-21) demonstrates that the reproducibility of the experiments is adequate. Weight increase is in mg/sq cm. Multiplying by 2.9 converts this to microns of metal oxidized and by 6.4 to microns of oxide formed.

Oxidation curves for ten Type 302 (18Cr-8Ni) specimens in wet and dry air at 1600, 1800 and 2000 F are shown in Figure 1. All but No. 8 were electropolished. The early parts of the curves are plotted on a more sensitive scale in the inset. There it can be seen that specimens 3, 4, 5 and 8 exhibit an initial short protective period of one to ten hours before embarking on their inherent scaling pattern; even specimens 6 and 7 show such a hesitation for 15 minutes despite the fact that wet air at 2000 F is too aggressive for 18/8 and the scale never again becomes protective. The initial extra protectiveness is due to a thin oxide of special composition formed prior to the scaling runs and during the heating up period. The unusual nature of curves 9 and 10 whose slope increases steadily from 100 hours on is associated with the growth of local eruptions on the scale; these warts were first detected between 100 and 125 hours.

Figure 2 shows the oxidation curves for seven Type 330 (15Cr-35Ni) electropolished specimens in wet and dry air at 1800 and 2000 F.

Figure 3 shows the rate curves for four Type 309 (25Cr-12Ni) samples in wet air at 2000 F and illustrates the effect of three preliminary treatments: No. 18 was electropolished, No. 19 abraded, and Nos. 20 and 21 abraded and brought to temperature in helium. The somewhat lower scaling rate of Nos. 20 and 21 for the first 50 hours is believed due to a preferentially oxidized film high in chromium formed during the preheating in helium; in spite of being purified by activated charcoal at liquid nitrogen temperature the helium was somewhat oxidizing because of contamination from the hot ceramic furnace tube and packing.

TABLE 1—Composition of Stainless Steels (Weight Percent)

STEEL	C	Mn	Si	Ni	Cr
A. Type 302 (Spec. 1 & 2)	0.11	0.34	0.59	8.3	19.0
B. Type 302 (Spec. 3 to 9)	0.07	1.49	0.40	10.1	18.7
C. Type 330	0.06	1.66	0.65	35.0	14.3
D. Type 309	0.12	1.90	0.63	13.2	23.0

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TABLE 2—Summary of Oxidation Runs and Examination of Scales

Temp, Degrees F	Alloy	WET AIR Spec.	Pretreatment	Time, Hrs.	Wt. gain[1] mg/sq cm/ day at 100 Hrs.	X-Ray Diffraction[3] Spinel	Rhomb.	Other	Chem. Anal.[6] Fe	Cr	Ni	Mn	Si
1600	302 (A)	1	Electropolished	74	(0.54)[2]	m 8.34[4]	m (Fe)[5]		55	9	4	0.3	0.1
						m 8.34	m (Fe) s (Cr)		53	10	3	0.4	1.5
1800	302 (B)	3	Electropolished	53	(> 4)								
		4	Electropolished	52	(> 3)	s 8.35	l (Fe)		60	3	6	0.2	0.2
						l 8.34	s (Cr)		50	13	6	0.2	1.0
2000	302 (B)	6	Electropolished	9	(> 20)								
		7	Electropolished	12	(> 20)	m 8.39	m (Fe)		63	3	2	0.1	0.2
						l 8.40		Metal					
		8	Abraded, helium	25	(> 20)								
1800	330 (C)	11	Electropolished	581	0.30				33	22	10	2	0.8
		12	Electropolished	1034	0.29	l 8.43	s (Fe) s (Cr)						
						l 8.43	s (Cr)	Metal					
						l 8.43	s (Cr)	Metal, silica					
2000	330 (C)	14	Electropolished	648	0.64	l 8.46	s (Cr)						
						m 8.44	m (Cr)	Metal	34	17	12	6	0.8
						m 8.44	m (Cr)	Silica					
		15	Electropolished	723	0.81	l 8.38							
						l 8.37	s (Cr)						
						m 8.44	m (Cr)	Metal	35	10	16	6	1.0
						s 8.44	l (Cr)	Silica					
2000	309 (D)	18	Electropolished	722	0.65								
		19	Abraded	243	1.4								
		20	Abraded, helium	241	0.89								
		21	Abraded, helium	408	0.71								

Temp, Degrees F	Alloy	DRY AIR Spec.	Pretreatment	Time, Hrs.	Wt. gain[1] mg/sq cm/ day at 100 Hrs.	X-Ray Diffraction[3] Spinel	Rhomb.	Other	Chem. Anal.[6] Fe	Cr	Ni	Mn	Si
1600	302 (A)	2	Electropolished	361	0.16		l (Fe)[5] s (Cr)		51	17	1.0	1.5	0.4
									65	4	..	..	1.2
1800	302 (B)	5	Electropolished	504	0.69	s 8.45[4]	m (Fe)		49	18	1	1.5	0.2
						m 8.45	s (Fe)		52	14	2	1.3	1.0
							l (Cr)		54	14	..	1.0	1.0
									62	5	..	..	3
2000	302 (B)	9	Electropolished	265	0.96	m 8.45	m (Fe)		49	15	4	1.5	0.2
						l 8.45	s (Fe)		48	16	4	1.5	0.2
						m 8.44	m (Cr)		46	20	3	1.5	0.8
		10	Electropolished	340	0.92								
1800	330 (C)	13	Electropolished	505	0.34	l 8.42	s (Fe)	Silica	56	9	3	2	0.8
						l 8.43	s (Cr)		52	13	4	0.5	1.5
2000	330 (C)	16	Electropolished	391	0.65	l 8.44	s (Cr)						
						m 8.44	m (Cr)						
						m 8.44	m (Cr)	Silica					
		17	Electropolished	385	0.72								

[1] Since run duration varies, weight gain has been calculated in mg/sg cm/day for all specimens at 100 hours to show the relative protectiveness of the scales.
[2] Weight gains in parentheses are estimated projections to 100 hours for runs of shorter durations.
[3] Data for different layers are listed in order, the outermost layer at the top. The relative amounts of the phases are indicated by l, m & s, (large, medium and small).
[4] Spinel parameter is in Å.
[5] (Fe) indicates lattice parameter of M_2O_3 phase is closer to Fe_2O_3;
(Cr) indicates lattice parameter of M_2O_3 phase is closer to Cr_2O_3.
[6] The analyses are approximations obtained by combining the spectrographic and colorimetric results of a number of sample fractions.

Nearly all the curves exhibit one or more breaks; thus the abrupt changes in scaling rate observed with the chromium steels[1] appear to extend to stainless steels generally. All Type 330 specimens—1800 or 2000 F, wet or dry air—show a break around 2mg/cm², as do three of the Type 309. Perhaps this represents a critical oxide thickness for a certain set of experimental conditions. Breaks in the same weight gain range have been reported previously.[3, 4, 5]

The data have not been treated to deduce whether the kinetics of the scaling reaction conform to parabolic or to other rate laws. On the proper coordinates, sections of the curves yield reasonably satisfactory straight lines. Little is gained, however, beyond confirming that at intervals the process is diffusion controlled and is consistent with a Wagner type of mechanism. For much of the time this diffusion mechanism is obscured by other effects such as separation at the metal/scale interface and local eruptions.

Effect of Moisture in Air

Moisture in the air has an intense effect on the scaling of Type 302 (Figure 1). In wet air at 2000 F, scaling proceeds very rapidly (specimens 6 and 7) whereas in dry air a highly protective scale is maintained for many hours (Nos. 9 and 10). A similar relationship is evident at 1800 F and 1600 F. The scale is even less protective in wet air at 1800 F than in dry air at the higher temperature of 2000 F (3 and 4 vs 9 and 10). This is notably the reverse of what was observed with Type 446 steel where scaling was more severe in dry air, although less severe in dry air with Type 410 and 430.[1] For Type 330 the moisture effect is less marked: wet and dry are equivalent at 1800 F and wet air is more aggressive than dry at 2000 F. The effect of moisture would appear to depend sensitively on alloy composition, that is, on scale composition.

The results of X-ray diffraction and chemical analysis are presented in abridged form in Table 2. The two major constituents of the scales are spinels and rhombohedral oxides. The spinel, M_3O_4, is capable of wide variation in composition as indicated by the general formula:

$$(Fe, Ni, Mn)\ O.\ (Fe, Cr, Mn)_2\ O_3$$

The rhombohedral oxide, M_2O_3, also may have variable composition over the range $(Fe, Cr, Mn)_2O_3$. Silica and elemental metal are minor constituents. Silica occurs at the metal/scale interface, below the surface as a subscale, or between two scale layers. Metal occurs in small amounts as the residue of severely intergranularly corroded grains and in large amounts in rapidly growing scales and the inner scale of warts.

For ease of interpretation the results are presented as a qualitative summary. A more rigid presentation would be of little benefit since a number of factors preclude unambiguous correlations. The relation between composition and spinel lattice parameter, established for the Fe-Cr spinel solutions,[6, 7] is unknown for additions of Mn and Ni and is not predictable other than that Mn would raise the parameter and Ni lower it. Similarly, the presence of the third metallic element, Mn, prevents use of the observed lattice parameter to deduce the composition of the rhombohedral phase, although, again, this is known for the simple $(Fe, Cr)_2O_3$ solutions.[8] As a result, since the scale layers are usually intimate unseparable mixtures of the different phases and hence the chemical analysis must represent only an over-all composition, it cannot be known in a particular layer whether, say, a high Mn content should be assigned to the spinel or rhombohedral phase.

Contributing to the complexity is the fact that the scale composition varies not only with depth but from one area to another; warts are the extreme of this condition. For those reasons, in Table 2 the relative amounts of M_3O_4 and M_2O_3 are expressed only as large, moderate or small; the spinel parameter is given to the nearest 0.01Å; the M_2O_3 parameter is described simply as being closer to that of Fe_2O_3 or Cr_2O_3; the chemical analyses are approximations—combinations of spectrographic and colorimetric results of a number of sample fractions. In spite of these difficulties there is reasonable correlation in the data; for instance, when the Mn content is high or the Ni low the spinel parameter is high and vice versa.

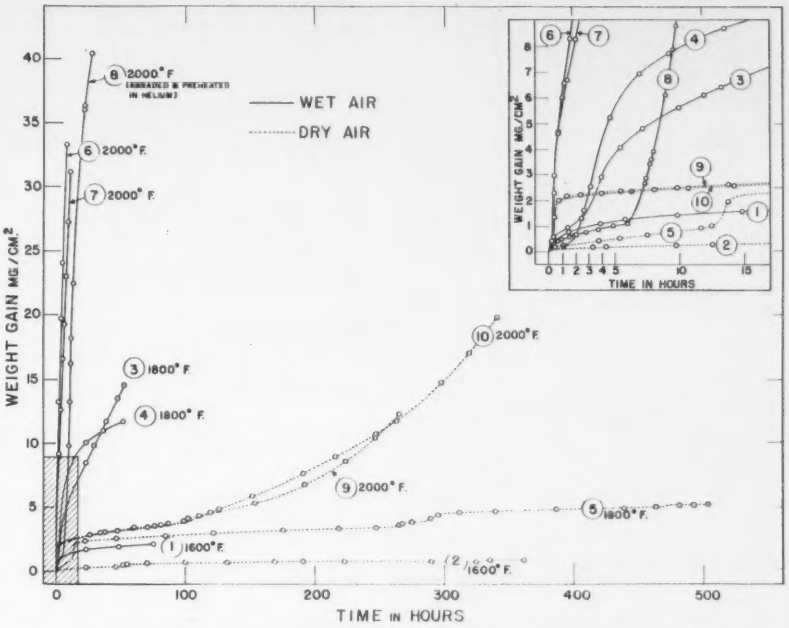

Figure 1—Oxidation of Type 302 stainless steel in wet and dry air at 1600, 1800 and 2000 F. All samples were electropolished except specimen 8 which was abraded (and brought to temperature in helium).

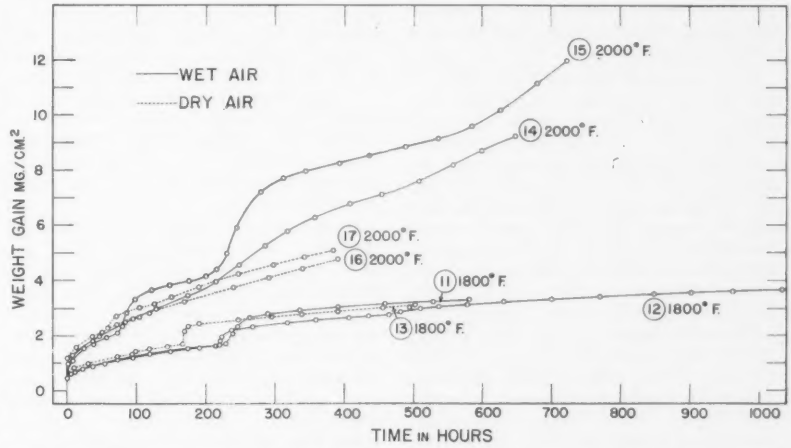

Figure 2—Oxidation of Type 330 stainless steel in wet and dry air at 1800 and 2000 F. All samples were electropolished.

Preferential Oxidation Effects

In general the analyses demonstrate the expected preferential oxidation effects. The extent to which Si, Mn and Cr increase and Ni decreases in the

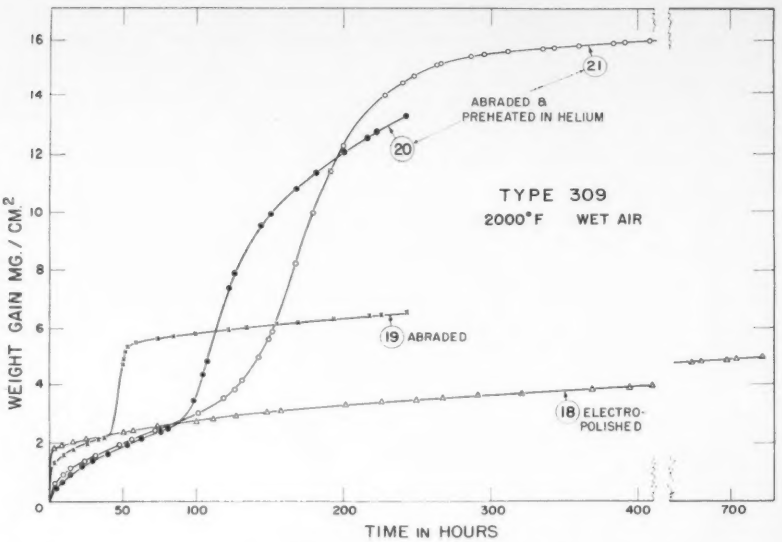

Figure 3—Oxidation of Type 309 stainless steel in wet air at 2000 F. Specimen 18 was electropolished, No. 19 abraded, Nos. 20 and 21 abraded and brought to temperature in helium.

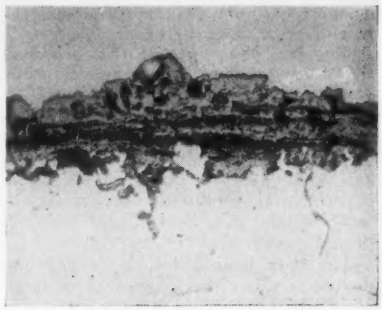

Figure 4—Metallographic section of scale formed on specimen 16 (Type 330) after 391 hours in dry air at 2000 F. Subscale islands and intergranular stringers are partly silica. Unetched, 600X.

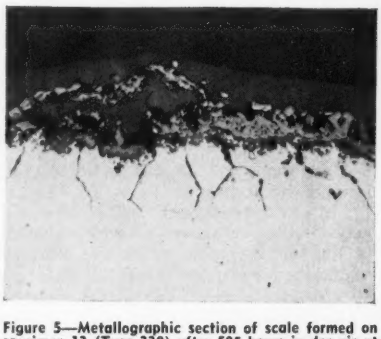

Figure 5—Metallographic section of scale formed on specimen 13 (Type 330) after 505 hours in dry air at 1800 F showing an outer layer which presumably separated from the metal at the 170 hour break of curve 13. Note metal islands in scale close to metal surface. Unetched, 500X.

scale depends on the scaling rate and scale thickness. Cr is higher in the more slowly scaling specimens but the outermost layer of all scales is lower in Cr due apparently to the lower diffusivity and perhaps also to the higher volatility[1] of Cr compared to Fe.

The innermost layers of specimens 2 and 5, shown in Table 2 to be especially high in Fe and Si, are not really samples of scale but of a film of interference color thickness which formed under the silica layer on areas of the metal exposed on cooling as the scale flaked off. The high Si content thus reflects the presence of a silica layer at the metal/scale interface, and the high Fe the fact that the surface metal was depleted in Cr under the scale. Appreciable Ni contents exist in the inner layers at high scaling rates and in the inner layer of warts. Here the high Ni is present both as finely divided metal phase and as a constituent of the spinel, as evidenced by spinel parameters as low as 8.32Å.

The Si accumulated at the metal/scale interface is not in as continuous a film as with the Fe-26Cr alloy,[1] but more as subscale islands and intergranular stringers (Figure 4). Silica shows up in the X-ray patterns of the innermost layer in a number of the Type 330 specimens and in the outer layer of No. 13 (Table 2). The latter can be related to the curve break at 170 hours in Figure 2 and to the separated outer layer visible in Figure 5. Segregation of Si at the metal/scale interface for Type 302 is demonstrated in nearly all the specimens by the chemical analyses (Table 2)—not, however, in the X-ray patterns. Silica is reported as occurring in an amorphous form in scales;[8,9] perhaps the time-temperature combination in the Type 302 experiments was not sufficiently intense to develop adequate crystallinity in the SiO_2 whereas it was with Type 330.

Figures 6 to 10 illustrate some points of interest from the metallographic examination. Note in Figure 6 that the outer light grey M_2O_3 layer and middle darker grey spinel layer of specimen 5 is succeeded by an inner light grey M_2O_3 layer (these are readily identified in polarized light). This suggests that, with the low scaling rate (0.08 mg/cm²/day) of the latter part of the run, insufficient Ni enters the scale to stabilize any spinel. Another possibility is that when a low scaling rate continues for a long time, Ni diffuses back to the metal substrate, allowing the oxide to change from spinel to rhombohedral.

Figure 7 and Table 2, specimen 2, show that at the still slower scaling rate of 0.007 mg/cm²/day no spinel appears. Figure 8 shows the structure of a moderate sized wart; only a few warts grew on this specimen. Figure 9 shows a type of small slowly growing wart which grew profusely on specimen 1, occupying perhaps 25 percent of the total specimen area. Figure 10 illustrates the mixture of oxide and finely divided metal, high in Ni, making up the inner layer of rapidly scaling specimens and rapidly growing warts.

Discussion

The accumulation of silica at the metal/scale interface, previously observed on Type 446 steel,[1] is seen to occur also with the austenitic stainless alloys. The authors believe that, as with Type 446, the silica film contributes directly to the breaks in the rate curves. Specimen 13 (Figure 5) is an example of this association: silica is identified between two separated scale layers while the thickness of the outer, first-formed layer is consistent with the weight gain up to the break at 170 hours (Figure 2). The silica segregates partly as a subscale rather than exclusively as the continuous films of Type 446, perhaps because of a higher solubility of oxygen in austenite. This would explain why there is less correlation between silica and breaks in the curves for the austenitic steels and suggests that additional factors contribute to the break mechanism.

Type I Break

A distinction between two types of breaks should be made. Type I marks the end of an initial period of extraordinary protectiveness resulting from a thin oxide film which is very high in chromium. This film is formed from a combination of the surface preparation procedure and preferential oxidation taking place while heating to test temperature or in an atmosphere of low oxidizing potential. (Whether Si contributes to the protectiveness of the thin oxide is not yet clear.) This phenomenon was seen very strikingly with the Fe-Cr alloys (Figure 4 of reference 1) where negligible weight gain occurred for as long as 240 hours, after which the specimens oxidized at the rapid rate normal to the alloy. Type 302 specimens Nos. 3, 4, 5 and 8 show a similar behavior (Figure 1 inset). The lower initial scaling rate of Type 309 specimens Nos. 20 and 21 compared to 18 and 19 is a related effect caused by preferential oxidation in an impure helium atmosphere, but the temperature is too high for the establishment of the very slow rates.

An initial period of marked protectiveness has been reported by several workers, being described as an induction period,[10,11,12] breakthrough,[3] or otherwise.[13,14] Perhaps more importance than is warranted has been attached to this effect since it reflects the method of surface preparation and pretreatment and

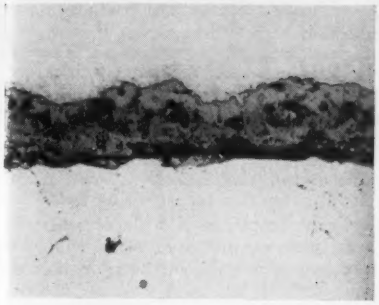

Figure 6—Section through scale formed on specimen 5 (Type 302) after 504 hours in dry air at 1800 F. Note middle layer of spinel (darker grey between two layers of rhombohedral oxide (lighter grey). Unetched, 600X.

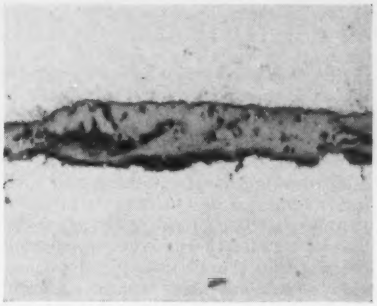

Figure 7—Section through scale formed on specimen 2 (Type 302) after 361 hours in dry air at 1600 F. There was no isotropic oxide (i.e., no spinel). Unetched 1000X.

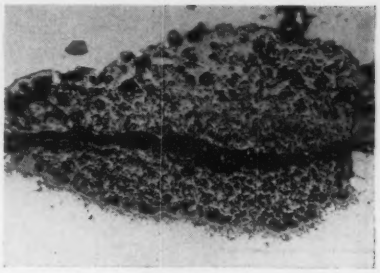

Figure 8—Section through wart on specimen 16 (Type 330) at 2000 F in dry air. Unetched, 250X.

not the intrinsic oxidation characteristics of the alloy composition.

Mechanical rupture has been suggested as one explanation of the thin-film break.[14] But the fact that the film is thin and presumably sufficiently plastic to remain in tight contact with the metal makes it doubtful that a crack would expose the metal to the oxidizing gas at other than the root of the crack. A more probable mechanism is one in which the high Cr content of the oxide is diluted by transfer of cations from the alloy. McCullough, Fontana and Beck[14] found that the Cr content of the thin oxide before the break was over 60 percent, or 90 percent of the metals present. This high Cr content, the result of preferential oxidation, yields a scale through which cations diffuse extremely slowly. As Fe, Mn and Ni ions invade the scale, the composition is diluted to one tolerating a higher concentration of cation vacancies and through which cations diffuse more rapidly. It follows that only one Type I break is possible per specimen and early slow scaling rate is seldom regained. The break need not appear; under the same conditions of surface preparation and pretreatment the probability of an initial slow rate occurring is highest at the lower temperatures.

Type II Break

The second type of break is one which occurs after a relatively thick (greater than 10 μ) protective scale has formed. Such breaks are shown by nearly all the Type 330, 309, 446 curves and by some of the more slowly scaling Type 302. After the break the scale becomes protective again and scaling continues at a rate similar to that before the break. Type II breaks can occur repeatedly on the same specimen.

The nature of the Type II break suggests it is caused by cracking of the scale and oxygen being admitted to a large area of the metal. This would appear to require considerable loss of contact between scale and metal, otherwise only the small area at the bottom of the crack would be affected.

The authors believe that formation of voids and silica at the metal/scale interface both contribute to this separation. Separation by itself impedes cation transfer and results in a very slow scaling rate. Cracking of the scale is therefore necessary for the break to occur. The time at which this takes place depends on more than one factor: the extent of separation (i.e. the amount of silica plus voids), the extent to which the scale is keyed to the metal by interlocking peninsulae of scale and metal, the thickness of the layer, and, related to this, the stress level in the scale.

Stress is produced or heightened by volume changes associated with recrystallization. The partial insulation of the scale from a fresh cation supply allows its state of oxidation to increase, causing spinel to change to rhombohedral or to rhombohedral plus spinel of different composition (higher in Ni). Such lattice changes plus the appreciable loss of contact with the metal encourage the scale to crack free over a large area. The surface layer of metal will be depleted in Cr, Si and Mn[15] (to an extent depending on the scaling history—a long time at slow rate allows the surface metal to be replenished in alloying elements from the body of the specimen) so that the curve after the break can be very steep until a new film of appreciable thickness builds up.

Under the present experimental conditions, breaks (Type II) become the general rule; a specimen not showing a curve break is then abnormal. (The alloy content of Type 302 is too low for its scaling behavior to fit completely into this general pattern.) Thus it seems necessary to seek an explanation for the absence of a break in curve 18.

Vermilyea[16] has recently presented an illuminating theory of cation transfer whereby voids need not develop at the interface since the lattice vacancies generated by movement of cations from metal to oxide may be continuously destroyed by a sidewise movement of lattice steps along the surface—analogous to dislocation climb in a crystal. But this mechanism applies mainly to thin films at moderate temperatures. With thicker films the plasticity would be insufficient for them to conform even by Vermilyea's mechanism to the retreating metal, and voids must form (assuming that oxide growth is predominantly by cation diffusion).

MacKenzie and Birchenall's experiments on the plasticity of iron oxides,[17] in which they found that only FeO was sufficiently ductile not to generate a cavity on the metal on which it grew, lead one to expect that the spinels and rhombohedral oxides on stainless steel are too inflexible not to form voids. How is it possible then to account for the absence of a break in curve 18? Either (1) The scale is relatively plastic so that void formation is depressed, (2) Silica segregates less at the interface (which appears a fact since much subscale is observed in Type 309 specimens), or (3) Because of the scale composition, lattice changes did not develop enough stress to crack the scale. The latter suggestion seems the most reasonable. The difference between specimen 18 and the others is one of degree only since breaks did occur when the same alloy was not electropolished. Even No. 18 would be expected to break if the run had been further extended.

Growth of Warts

The factors involved in the growth of warts are not properly understood. Perhaps they initiate at the root of a scale crack for the case where loss of contact between metal and scale is not enough to yield the Type II break. But one would expect the crack to heal itself quickly. Instead, the wart grows rapidly even when many times thicker than the scale proper, as though growth were by surface or grain boundary diffusion, not lattice diffusion. Or perhaps warts initiate at the contact points between scale and metal after extensive loss of contact has taken place. The Ni content is high in warts but it is uncertain whether this is the cause or effect of the high local scaling rate.

Effect of Moisture

An adequate explanation of the effect moisture in air has on the scaling process has not yet been expressed. Kubaschewski and Hopkins[18] summarized much of the data available, citing cases in which moisture increases and decreases scaling rate, but point out that no systematic investigation has yet been reported. Previously it was suggested[1] that by occupying cation sites in the scale, hydrogen ions might decrease the protectiveness of M_2O_3 high in Fe, and increase that of M_2O_3 high in Cr. The latter was proposed to explain the inhibitive effect of moisture on the scaling of Type 446.

On the other hand, David and Welch hold that OH^- can be expected to substitute for $O^=$ in the oxide lattice[19] in

Figure 9—Section through one of numerous small warts liberally covering this slowly scaling specimen 1 (Type 302). Unetched, 1000X.

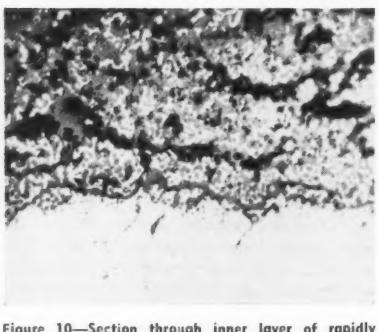

Figure 10—Section through inner layer of rapidly growing scale on specimen 7 (Type 302). Sample was exposed 12 hours in wet air at 2000 F. Note finely divided mixture of metal and oxide. Unetched, 500X.

which case a larger number of cation vacancies can be compensated for and hence an increased rate is to be expected, as observed with the Ni-bearing alloys. A proper explanation must account for the extremes observed in the moisture effect: inhibition with Type 446, acceleration with Types 410, 430 and 302, and little or no effect with Type 330.

Effect of Prior Treatment

The marked effect of surface preparation and pretreatment, shown in Figure 3 and curve 8 of Figure 1, has been observed many times.[20-23] Kubaschewski and Hopkins (reference 18, page 72) summarized the work up to 1952. Except in some special short time applications such as rocketry, it is the long term heat resistance of an alloy which is important. The objective in any surface preparation, therefore, should be to leave as innocuous a surface film as possible. An actual film-free surface cannot be achieved with active metals such as here discussed.

Work is now in progress in this laboratory in which metals prepared by various abrading, polishing and etching methods are being examined by electrochemical and electron diffraction techniques. The results demonstrate that the surface is sensitive in a complex fashion to modification by even mild treatments. Redeposition of one constituent of an alloy during electro-polishing and etching is found to be much more prevalent than expected. On the evidence now developing, many surface preparations described in the literature and presumably still being used, appear deficient, unless perhaps for very long runs. Specifically, the resultant defects are embedded abrasive, preferential oxidation, preferential etching, and redeposition of metal; the surface stress and roughness factor may be of lesser importance. Preliminary work indicates a preferred preparation to be electropolishing followed by the shortest possible etch that will remove the electropolishing film.

The foregoing would indicate that serious gaps exist in the understanding of alloy scaling, with the exception perhaps of idealized systems.[24] The additional variables resulting from the presence of the several minor constituents in commercial stainless alloys further complicate the system. This has contributed to the difficulty of deducing unequivocal correlations between the experimental data and a scaling mechanism. When, elsewhere, such interpretations have been attempted—some have been most ingenious and elaborate—it has been with the assumption that additional variables beyond those considered are minor in effect. For example, Yearian et al point out that much of their interpretation is necessarily speculative.[25] For these reasons the authors' current research in alloy scaling is being conducted with specially prepared pure binary alloys. Additions of a third constituent permit its specific effect to be investigated. Using such test materials in well designed experiments, and paying adequate attention to surface preparation, it is expected that data capable of unambiguous interpretation will be obtained.

Summary

Breaks in the rate curves appear to be a general phenomenon in the scaling of stainless steels. They occur in Type 302, 330 and 309 as well as in the Type 410 430 and 446 previously observed.

Two types of break may be distinguished. Type I marks the end of an initial period of extraordinary protectiveness caused by a thin film of high Cr (and perhaps Si) content formed during the prior treatment given the specimens. Modification of the film composition by dilution with other cations destroys the high degree of protectiveness. Type II breaks occur after a relatively thick protective scale has formed. Isolation of the scale from the metal by accumulation of silica and voids at the metal/scale interface, followed by cracking of the scale due to recrystallization, exposes the bare metal surface to the oxidizing gas.

Local eruptions of scale or warts are a common occurence on stainless steel. A proper explanation of the growth is not yet forthcoming.

Moisture in the air, previously observed to decrease the scaling rate of Type 446 and increase that of 410 and 430, increases the rate of the Ni-Cr steels also. The effect of moisture might be to increase the concentration of cation vacancies through OH^- substituting for $O^=$ in the oxide lattice, or to decrease it by H^+ substituting for Fe^{++} or other cation. An adequate theory, however, is still to be evolved.

Two factors requiring closer attention in scaling investigations are methods of surface preparation and a decrease in the extra variables introduced by minor constituents present in many alloys.

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Any discussions of this article not published above will appear in the June, 1959 issue

Comparative Corrosion Resistance Of 200 and 300 Series Stainless Steels In Chemical Manufacturing Processes*

By A. C. HAMSTEAD and L. S. VAN DELINDER

Abstract

Data are presented showing the comparative corrosion resistance of several experimental heats and of two commercial heats of AISI 200 series chromium-nickel-manganese steels with the 18-8 chromium-nickel steels (300 series) in a variety of chemical manufacturing processes.

Results of this preliminary comparison have indicated that the chromium-nickel-manganese alloys may be utilized in a number of chemical services where the higher nickel compositions (18-8 steels) are now used. Some environments were encountered in chemical plant processes in which the chromium-nickel-manganese steels in the annealed condition were more corrosion resistant than the 300 series (Types 304 and 316).

In some of the plant processes tested, it was found that in the sensitized condition (1-hour at 1200 F), the susceptibility of the commercial heats of 201 and 202 to intergranular attack was greater than that of the 18-8 chromium-nickel steels. 6.25

Introduction

THERE HAS been increased interest during the past decade in chromium-low nickel-manganese steels. This was brought on in part by the acute shortage of the world's supply of nickel and the ensuing restriction imposed by government M-80 regulation limiting the nickel content to 1 percent for civilian uses. Detailed and comprehensive studies have been made within this period to arrive at compositions of low-nickel steels that would have an austenitic structure, mechanical properties, and corrosion resistance comparable to the familiar 18 chromium-8 nickel stainless steel alloys.[1,2]

From these studies a "new family" of austenitic stainless steels has been developed and subsequently adopted as standard materials by the American Iron and Steel Institute. These chromium-low nickel-manganese compositions, which have been developed primarily to conserve nickel, are known as the AISI "200 series."

Two of these materials, 201 and 202, already are being produced commercially in increasing tonnages (less than 2000 tons in 1955 to 19,400 tons in 1956),[3] and it is expected that eventually these materials will replace 80 percent of their higher-nickel counterparts, 301 and 302.[4] These new steels (201 and 202) are gaining acceptance by the transportation, automotive, home appliance, restaurant, and architectural industries, and to an appreciable extent by the dairy and brewing industries. Reports compiled by the Committee of Stainless Steel Producers have led to the conclusion that: (1) These steels can be produced and fabricated without appreciable modification of the techniques employed for the 18-8 stainless steels, (2) These steels can replace Types 301 and 302 without limitation for all services where the latter steels are being properly used, and (3) At elevated temperatures, the short time tensile and creep-rupture properties of Types 201 and 202 are superior to their counterparts of the "300 series" and compare favorably with those of Type 347.[5]

Although the position of the 200 series steels now appears to be well established with respect to Type 301 and 302 stainless steel, much interest has developed in establishing their position for more corrosive services in industries where Types 304 and 316 are now utilized in large tonnages. Soon after adoption of the 200 series as standards, the Department of Defense appealed to all industries to make the "fullest use of the substitute materials" to enable the replenishment of our stockpile of nickel.[6] On May 1, 1956 through the joint efforts of the Chemical Industry Advisory Board and the AISI Stainless Steel Technical Committee, a program was launched to implement a plan of action to encourage "the use of low-nickel and nickel-free stainless steels to achieve their early and widespread applications in the place of the 300 series."[7] Details of the CIAB and AISI program may be procured from the International Nickel Company who will also furnish test samples at no cost and act as a clearing house for the corrosion data gathered from the various chemical companies.

The steel producers have compiled and made available much information on compositions of the chromium-low nickel-manganese steels which are most likely to prove successful throughout all industries. All the same, much information must yet be obtained on the corrosion resistance of these new steels in laboratory tests, field tests and service performance before they will receive acceptance by the chemical industry for the more corrosive services where Types 304 and 316 are in so much demand. Energetic

Van Delinder

Hamstead

L. S. VAN DELINDER is a group leader of Materials of Construction Group, Development Department, Union Carbide Chemicals Co., South Charleston, W. Va. His 19 years of corrosion control experience includes most types of organic and inorganic corrosion problems encountered in chemical plants. Mr. Van Delinder has a BS in chemistry from Morris-Harvey College. He is chairman of Technical Unit Committee T-6 on Protective Coatings and is active in other NACE technical committees.

A. C. HAMSTEAD is staff associate, Development Department, Union Carbide Chemicals Company, South Charleston, West Va. He received an AB in chemistry from West Virginia University in 1930 and has more than 20 years experience in plant corrosion and metallurgical problem. He is a member of NACE, ASTM and ASM.

TABLE 1—Alloys Used in Corrosion Tests

ALLOY	ELEMENT							
	Carbon	Chromium	Nickel	Manganese	Silicon	Sulfur	Phosphorous	Nitrogen
GM-82	0.05	18.07	4.97	4.97	0.57	....	....	0.14
GM-83	0.05	18.27	5.21	5.23	0.71	....	....	0.12
R-975	0.05	17.74	4.21	8.54	0.42	....	....	0.17
R-976	0.09	17.69	4.46	8.93	0.40	....	....	0.17
201	0.06	17.44	4.62	6.68	0.33	0.010	0.025	0.175
202	0.09	17.68	5.03	9.35	0.50	0.013	0.030	0.15
204L	0.024	17.22	5.39	9.99	0.64	0.010	0.024	0.20
304	0.071	18.95	8.96	0.43	0.45	0.018	0.017	....
201 (Nominal)	0.15 Max.	16–18	3.5–5.5	5.5–7.5	....	....	....	0.25 Max.
202 (Nominal)	0.15 Max.	17–19	4–6	7.5–10.0	....	....	....	0.25 Max.

★ Submitted for publication November 18, 1957. A paper presented at a meeting of the South Central Region, National Association of Corrosion Engineers, Oklahoma City, Oklahoma, October 1-4, 1957.

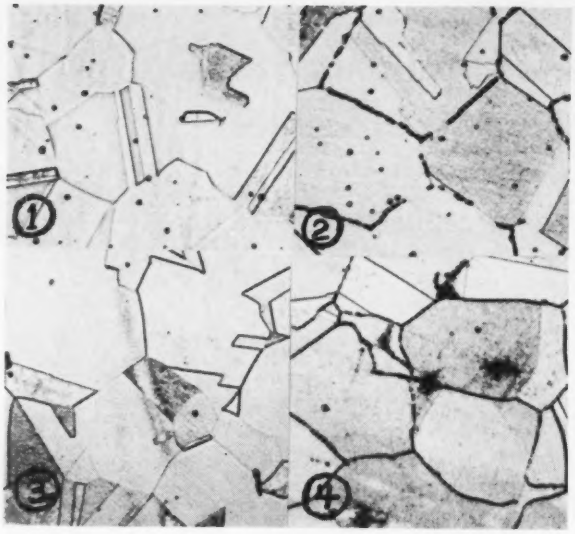


Figure 1—Microstructure of experimental chromium-nickel-manganese steels. Oxalic acid etch, 250X.
(1) R-975: heat treated 1970 F, 15 minutes, air-cooled.
(2) R-975: heat treated 1 hour at 1200 F, air-cooled.
(3) R-976: Same as 1.
(4) R-976: Same as 2.

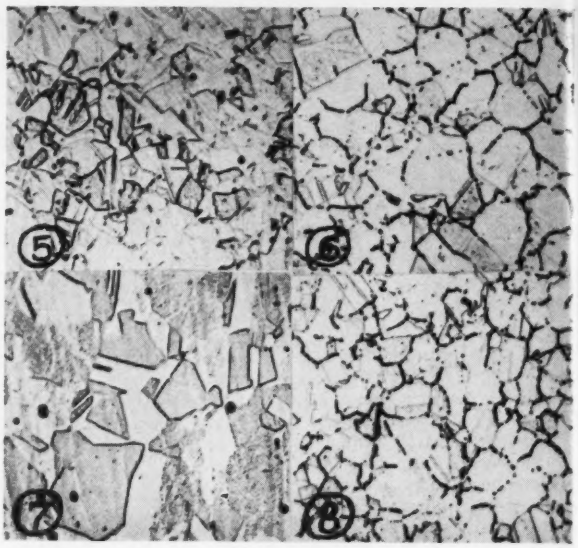


Figure 2—Microstructure of commercial heats of 200 series steels. Oxalic acid etch, 250X.
(5) 201—Annealed condition.
(6) 201—Sensitized condition: 1 hour at 1200 F, air-cooled.
(7) 202—Annealed condition.
(8) 202—Sensitized condition: 1 hour at 1200 F, air-cooled.

pursuit of corrosion testing programs and service performance tests by corrosion engineers in the petroleum and chemical industries will be required to determine the "range of service" in these industries for which the new steels will be suitable.

The primary purpose of this paper is to collate the data obtained up to the present in various chemical processes on the corrosion resistance of the chromium-nickel-manganese steels as compared with that of the 18-8 chromium-nickel steels. Both experimental compositions of the chromium-nickel-manganese steels received for testing in 1952 and commercial heats of these alloys procured after their adoption as standards in 1955 are included in the study.

As will be observed in this initial report there are many details missing, and the need of a better organized plan of testing and comparison will be apparent. The shortcomings of this presentation may be explained by the fact that specimens were received at various times for testing, and were included with other specimens only when an occasion arose for routine corrosion investigations, without much consideration being given to selected environments. While the data have many shortcomings, it is hoped that their presentation may indicate some categorical environment wherein further investigation of these materials is warranted, and also environments wherein such studies may not be justified.

Materials Tested

The chemical analyses of the chromium-nickel-manganese steels used in the tests and of the 18-8 stainless steels are given in Table 1. Figures 1 and 2 show microstructures of certain alloys with varying heat treatments.

The experimental compositions of the chromium-nickel-manganese steels were from small laboratory heats of steel prepared by the Development Department of the Union Carbide Metals Company and are related to those heats reported by Franks, Binder and Thompson in 1954.[1] Specimens from the GM-heat series were placed on test in 1952. The R-heat series were received for testing in the early part of 1954.

The experimental materials received for the tests were in the form of small plates approximately 12 inches long by 4¼ inches wide by ⅛-inch thick. The test plates had been solution heat-treated 10 to 15 minutes at 1970 F, air-cooled and then pickled.

As will be observed, the GM heats were within the nominal composition range of AISI Type 201 except that they were slightly low in manganese content, and one of them (GM-83) contained a small amount of copper. The R heats were both within the nominal composition range of AISI 202, differing only with respect to carbon content, R-975 having 0.05 percent carbon and R-976 having 0.09 percent carbon.

Subsequent to the adoption of the 201 and 202 grades, two standard mill sheets, one each of these two grades, were requisitioned for test purposes. These materials were supplied in the annealed and pickled condition.

Specimens of the 18-8 compositions tested were prepared from standard mill sheets, hot-rolled, annealed and pickled. These materials were selected from heats of steels meeting special requirements for optimum corrosion resistance.

Methods

To obtain further knowledge of the possible effects of fabricating techniques on general and intergranular corrosion resistance of the experimental and commercial heats of the steels, specimens were subjected to both the 65 percent nitric acid test,[8] representing strongly oxidizing conditions, and to the copper sulfate-sulfuric acid test.[9] These tests were conducted in accord with the ASTM Recommended Practice A262 and A393 respectively for the two tests.

For the field exposures in various plant process equipment, metal specimens were machined to size from the experimental alloy test plates and from the standard size mill sheets in the case of the commercial heats of 201 and 202. The edges of all specimens were machined to avoid shear strain. In preparation for exposure, all specimens were cleaned with scouring soap and a soft brush, then rinsed in acetone, dried in a desiccator, and weighed on an analytical balance. They were then assembled on "test spools" of the conventional type often referred to and described in the literature.

The specimens were normally 2 inches by 2 inches in size with the thickness varying with that of the sheet from which they were cut. The specimen sizes, as well as that of the test assemblies, were of necessity quite variable to facilitate installation at different locations in process equipment.

After exposure, the specimens were cleaned for reweighing by scrubbing with a bristle brush using liquid soap. In special cases when corrosion was severe and heavy tenacious scale or deposits were present, the specimens were cleaned either by immersion in boiling 25 percent sodium hydroxide solution containing granulated zinc or in hot dilute nitric acid solution.

The results of a few laboratory tests are incorporated in the general corrosion data. To obtain these results, specimens were exposed under conditions simulating those of the plant processes as nearly as possible.

Each corrosion rate shown in the tables of data is the average of two to ten results obtained in that specific environment.

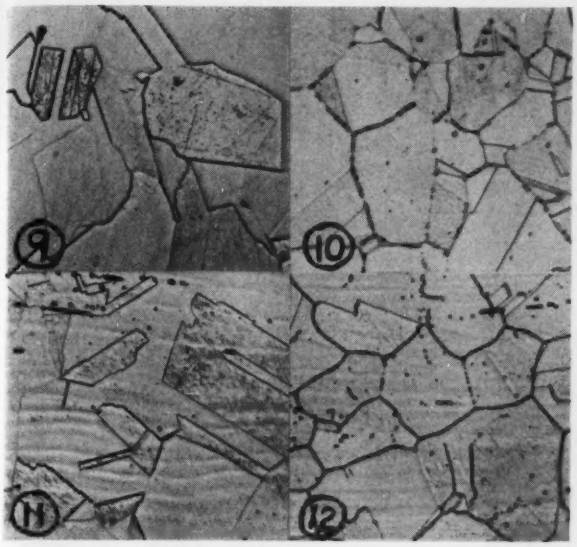


Figure 3—Microstructure of 204L and 304 steels. Oxalic acid etch, 250X.
(9) 204L—Annealed condition.
(10) 204L—Sensitized condition: 1 hour at 1200 F, air-cooled.
(11) 304—Annealed condition.
(12) 304—Sensitized condition: 1 hour at 1200 F, air-cooled.

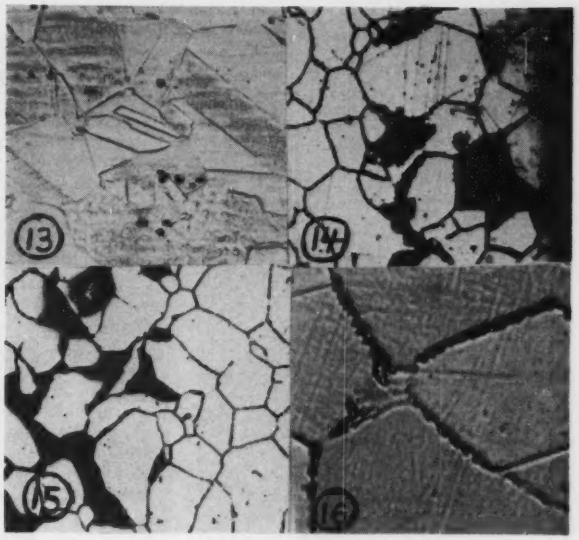


Figure 4—(13) GM-82—Heat treated 15 minutes at 1970 F, air-cooled.
(14) 202—Sensitized condition (after exposure in an organo-nitrile).
(15) 202—Sensitized condition (after exposure in weak acetic acid).
(16) 204L—Sensitized condition. See Figure 3 also. This precipitation is similar to that reported by Uhlig for nitrogen-bearing stainless steels.[32]
Oxalic acid etch, 250X except No. 16 at 1500X.

Discussion

Franks, Binder and Thompson[1] made a comprehensive study of the chromium-nickel-manganese steels containing nitrogen in an effort to determine composition ranges that would provide stable structures, suitable behavior in fabrication procedures, good mechanical properties, and good corrosion resistance. They found that steels containing 17 to 17.5 percent chromium, 3.5 to 4.5 percent nickel, 7 to 9 percent manganese, 0.06 to 0.12 percent carbon, and 0.12 to 0.18 percent nitrogen met the above requirements. These authors reported the corrosion resistance of these materials in strong oxidizing media, 65 percent boiling nitric acid (Huey Test), and in non-oxidizing media, air-free 10.0 percent sulfuric acid at 70 C.

Renshaw and Lula[2] have extended knowledge of the corrosion resistance of steels within the above composition range still further by reporting corrosion data in strongly oxidizing media and intermediate environments, and have reported upon the resistance of these materials to localized and intergranular attack. These authors also have described several compositions of the chromimum-nickel-manganese steels not yet having an AISI designation but with improved corrosion resistance for the more severe environments.

As indicated above, a large amount of laboratory corrosion data are available on various compositions of the chromium-nickel-manganese steels from the so called "evaluation tests" and from a limited number of solutions selected to represent conditions between those of strongly oxidizing and those of strongly reducing capacity. To the authors' knowledge, however, the only data available from field exposures of the chromium-nickel-manganese steels are those reported by the Corrosion Engineering Service of the International Nickel Company.[10] Some of the environments reported by INCO in which the corrosion rates of the chromium-nickel-manganese alloys appear to be the equivalent of Type 304 are acid fluorides, formaldehyde-methanol gas scrubbing media, sulfur distillations, liquid and vapor, "Bunker C" fuel oil-combustion condensate, and coal tar pitch constituents. Other environment in which the 200 series steels do not appear to be as resistant in data reported by INCO are sulfuric acid media and tar acids containing chlorides.

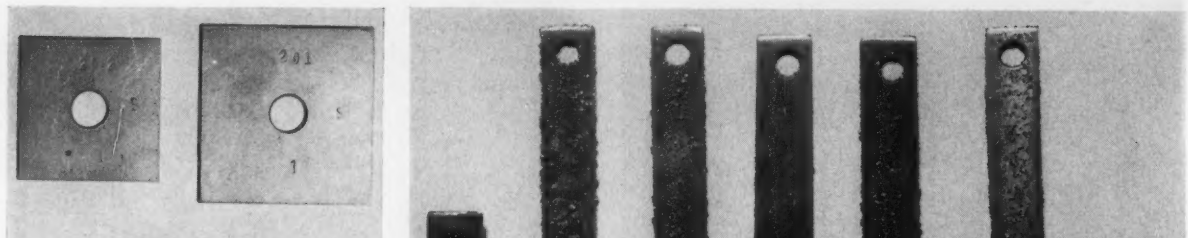
TABLE 2—Results of Nitric Acid and Copper Sulfate Tests on Chromium-Nickel-Manganese Steels

Metal	Boiling 65 percent nitric acid, period 1	2	3	4	5	ipm Avg. of 5	Copper Sulfate Test
	"As received" condition						
GM-82	0.0019	0.0016	0.0016	0.0016	0.0017	0.0017	Passed
R-975	0.0016	0.0016	0.0014	0.0016	0.0016	0.0016	Passed
R-976	0.0016	0.0015	0.0017	0.0016	0.0016	0.0016	Passed
Comm. 201	0.0014	0.0014	0.0015	0.0017	0.0016	0.0015	Passed
Comm. 202	0.0016	0.0018	0.0019	0.0023	0.0027	0.0021	Passed
204L	0.0018	0.0018	0.0018	0.0019	0.0023	0.0019	Passed
304	0.0011	0.0010	0.0009	0.0008	0.0028	0.0013	Passed
	Sensitized 1-hour at 1200 F, air cooled.						
GM-82	.....	.....	.....	.....	.....	.....	Failed
R-975	0.0260	.....	.....	.....	.....	.....	Failed
R-976	0.0785	.....	.....	.....	.....	.....	Failed
Comm. 201	0.0807	.....	.....	.....	.....	.....	Failed
Comm. 202	0.2225	.....	.....	.....	.....	.....	Failed
204L	0.0156	0.0089	0.0087	0.0101	0.0076	0.0098	Passed
304	0.0657	0.1175	0.1223	0.1071	0.0668	0.0958	Failed

Evaluation Tests

The evaluation tests were conducted to determine and compare the relative susceptibility of the steels tested to intergranular corrosion. Results of these tests are shown in Table 2. In the "as received" condition all of these materials passed the test. As was expected, these materials were severely corroded in the test after a sensitizing time of one hour at 1200 F. Likewise, the materials passed the Strauss Test in the "as received" condition, but all of them with the exception of the non-standard 204L composition failed to pass this test after the sensitizing time of one hour at 1200 F.

Regardless of the acceptable results obtained by exposure of the 204L alloy in the Huey and Strauss Tests, the appearance of this alloy containing 0.024 percent carbon was not entirely satisfactory after a sensitizing heat treatment for a short period (Figures 3 and 4). A definite carbide precipitation occurred at the grain boundaries although not continuous over all areas. Unfortunately, this material was procured too late to provide information on the effects of an extended field exposure of the metal in an environment where a uniform austenitic structure is required. The possible production of the low carbon grades of 200 series stainless steels is of great interest, and the



A B

Figure 5—(A) View of 200 series steels showing effect on ductility of metals in sensitized condition after exposure for various times in weak acetic acid. (B) View of sensitized experimental chromium-nickel-manganese steels and Type 304 showing embrittlement after exposure in Strauss Test.

TABLE 3—Corrosion Rates in Weak (30 Percent) Acetic Acid Storage

Heat Treatment	Corrosion rate, ipy			
	316	304L	202	201
	Ambient temperature–4000 hours			
As Received	Nil	Nil	Nil	Nil
Sensitized 1 hour at 1200 F, air-cooled..	....	Nil	0.001[1]	Nil[2]

	Test interval 1056 hours				
	316	304	204L	202	201
As Received	Nil	Nil	Nil	Nil	Nil
Sensitized 1-hour at 1200 F, air-cooled..	<0.000	<0.000	Nil	Nil[2]	Nil[3]

[1] Severe intergranular attack.
[2] Slight intergranular attack.
[3] Very slight intergranular attack.

TABLE 4—Tests in Acetic Acid-Acetic Anhydride Batch Still System*

	Corrosion Rate, ipy			
	316	304	202	201
	"As received" condition			
Kettle, 865 hrs.	0.006	0.003	0.006	0.008
Base of col, 1115 hrs....	0.003	0.013	0.020[1]	0.019[1]
Top of col, 2952 hrs.....	0.025	0.100	0.080[1]	0.165[1]
	Sensitized-one hour at 1200 F, air-cooled			
Kettle, 865 hrs.	0.003	0.005	0.024	0.015
Base of col, 1115 hrs.....	0.004	0.016	0.034[2]	0.032[2]
Top of col, 2952 hrs.....	0.020	0.097	0.091[1]	0.117[1]

* Temperature, maximum 155 C in kettle to maximum 130 C in top of column.
[1] Preferential attack along edges and at indentification marks.
[2] Pitted.

limitations in the use of such a material must be investigated.

Relative Resistance to Intergranular Attack in Various Chemical Processes

Inasmuch as resistance to intergranular corrosion must be taken into account for applications involving welding, stress relieving, or operation at high temperatures, data are given on the 200 series alloys in the sensitized condition in chemical plant environment. Standard practice for these tests was to heat treat the specimens one hour at 1200 F, air cool, and pickle to remove heat scale before exposure.

It is well known that the chromium-nickel-manganese steels, just like the chromium-nickel steels, are subject to intergranular attack when heated in the approximate temperature range of 1000 to 1600 F.

Binder, Thompson, and Bishop[11] found that these steels containing up to about 0.05 percent carbon, having 18 percent chromium with 14 percent manganese plus nickel content, and 0.20 percent nitrogen, were resistant to intergranular attack after one hour at 1200 F. Those containing 0.02 to 0.025 percent carbon and not more than 14 percent manganese plus nickel were resistant after 24 hours at 1200 F.

Renshaw and Lula[2] have compared the relative susceptibility of the chromium-nickel-manganese steels of varying carbon content from 0.017 to 0.10 percent after various sensitizing times at 1200 F. Based on Huey and Strauss tests, they concluded that the carbon content appears to be less critical in the chromium-nickel-manganese steels than in the chromium-nickel grades. The maximum carbon limits for the chromium-nickel-manganese steel at which intergranular corrosion does not occur after a sensitization heat treatment of 2 hours at 1200 F was established at 0.05 to 0.06 percent by these authors. This carbon limit for the 200 series steels corresponds to 0.03 percent for the 304 chromium-nickel alloy (304L).[2]

Some interesting observations were made on the relative susceptibility of the 200 series alloys to intergranular corrosion in the field tests along with the studies pertaining to their resistance to general corrosion. A few of these exposures are described in detail below.

In preliminary tests, the 200 series alloys appeared to be promising as substitute materials for 304 alloy for the storage of weak acetic acid. A new tank was needed for the storage of this acid (30 percent) at atmospheric temperature. Accordingly, tests were conducted under conditions of prolonged exposure to compare the resistance of the 200 and 300 series stainless steels in this service. Inasmuch as the tank was to be of welded construction, the metals were tested both in the "as received" and in the sensitized condition. The test assemblies were suspended in the storage tank in such a manner that they were completely immersed approximately 80 percent of the time and then in the vapor immediately above the liquid level 20 percent of the time. After 4000 hours the specimens were removed for evaluation. Table 3 summarizes the results of this test.

A photomicrograph showing intergranular attack on the 202 alloy in the sensitized condition after exposure in this

media is given in Figure 4. Figure 5A is a view showing the appearance of the 200 series specimens after exposure, and the effects of exposure on the ductility of the metals. There was no visible evidence of attack on any of the metals, and the weight losses were negligible. During the cleaning operation, it was observed that the 202 alloy in the sensitized condition had lost its metallic ring and could be bent in the hands much as a heavy piece of cardboard. Upon further examination it was found that this material had lost all of its ductility and was quite friable. The sensitized 201 alloy, although having lost a detectable amount of its metallic ring, was still quite ductile as indicated by a bend test (Figure 5A). The tests were repeated, but this time the specimens were removed after 1100 hours' exposure. The results were essentially the same as in the initial test. While the 202 alloy had lost its metallic ring in the second test of 1100 hours, it was still sufficiently ductile to be bent through 180 degrees without cracking.

The reason for the wide difference in the behavior of the commercial heats of the 200 series alloys probably is associated with the carbon content of the materials. It will be observed from Table 1 that the carbon content of the 202 alloy was 0.09 percent as compared with 0.06 percent for the 201 alloy. The manganese content of the 202 alloy was also appreciably higher than that of the 201 alloy, and it is possible that the higher manganese plus nickel content of the 202 alloy in conjunction with the higher carbon resulted in the greater susceptibility of this alloy to intergranular deterioration.[11] As expected, 304L and 304 in the sensitized condition were not susceptible to intergranular corrosion in the dilute acetic acid at ambient temperature.

The results of field test exposures in more severe acetic acid service are given in Table 4 and shown in the photograph of Figure 6. At some locations in this particular acetic acid-anhydride distillation process, the rates of attack were found to be significantly greater for the 202 and 201 alloys than for 304 alloy. Moreover, preferential attack on specimens along the edges and in and around cold-worked areas, support holes, and identification marks was decidedly more prevalent in the 200 series than in the 300 series alloys (Figure 6). Sensitized specimens of the 200 series were subject to incipient pitting attack whereas the 300 series alloys were not. In this particular environment again there was very little difference in the over-all corrosion rates or in the type and extent of attack on the 201 and 202 alloys.

In another field test which was conducted to determine whether existing equipment could be adapted to an entirely different service without excessive corrosion, data were obtained on the comparative corrosion resistance of Type 304 stainless steel and 202 alloy. In this process a cyclic diamine was being prepared in the presence of a catalyst under high pressure. Unfortunately, Type 304 in the sensitized condition was not included in this test for comparison. Nevertheless, the behavior of the 202 alloy to intergranular attack is again well demonstrated in this particular environment. Results of this test are given in Table 5. In the "as received" condition, 202 alloy was the equivalent of either Type 316 or Type 304 in this service. Here again the sensitized specimens had not undergone more weight loss than those in the annealed condition and were visibly unattacked. However, in six out of the eight test locations in the system, these specimens had lost their metallic ring, and at two of the locations tested, intergranular corrosion had proceeded to the extent that ductility of the metal was seriously impaired as indicated by a bend test. It was of passing interest to note that in one of the exposures, in fact the only one in which corrosion rates were appreciable, the 202 alloy in the sensitized condition apparently was not intergranularly attacked.

A field test of relatively short duration in the preparation of an organo-nitrile provided an interesting comparison of the corrosion resistance of the 200 and 300 series steels. The nitrile was being prepared by conversion of an organic cyanide in the presence of a suitable catalyst at moderately high temperatures. Difficulty was experienced with rapid failure of the Type 316 tubes in the hottest part of the converter and tests were conducted to find a more suitable material. The 200 series alloys were included in the tests as a matter of interest and for comparison only. The data of this test are shown in Table 6 and were interesting primarily because they disclosed a chemical environment in which Type 202 alloy in the annealed condition appears to have better resistance than the Type 304 stainless steel. The relative susceptibility to intergranular corrosion of the 200 series alloy and the 300 series (316 and 304) after a sensitizing heat treatment in this particular environment is again demonstrated in this test.

It should perhaps be mentioned that

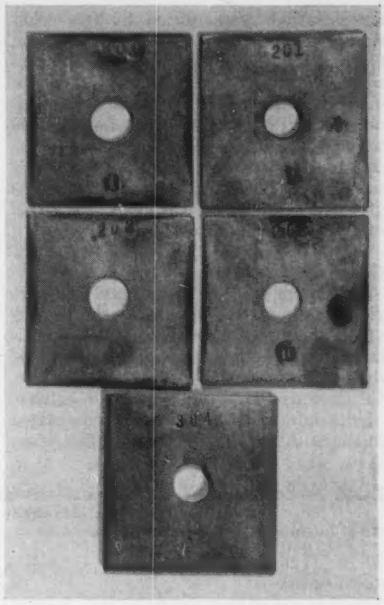


Figure 6—View of 200 series and Type 304 specimens after removal from an acetic acid-anhydride exposure showing intergranular etching and preferential attack on stressed areas of the 200 series alloys.

anomalous results were obtained in this test with respect to Type 304 in the "as received" and in the sensitized condition. Rates were much higher on this metal in the "as received" condition than they were in the sensitized condition. This does not frequently happen and the result was carefully checked by metallographic examination to determine if the specimens might have been reversed some way or another in the test. This was not the case, however, and the 304 specimens with the high corrosion rates were in the annealed condition while the ones corroded at the lower rates were in the sensitized condition.

Intergranular corrosion was again detected in the sensitized 202 specimens exposed in the distillation system where general corrosion was slight. These specimens had lost their normal metallic ring but had not yet lost ductility. The type of attack and microstructure of the 202 alloy after exposure to this media is shown in Figure 4. Sensitized Type 316 also had undergone slight intergranular

TABLE 5—Preparation of Cyclic Diamine at High Temperature and Pressure[1]

Metal	Location of Test Rack (Corrosion Rate, ipy)							
	Pre-heater	Surge Tank		Receiver		Recovery Still		
		Liquid	Vapor	Liquid	Vapor	Feed Tray	Liquid	Vapor
316	Nil	Nil	Nil	Nil	Nil	<0.001	0.004	Nil
304	<0.001	Nil	Nil	Nil	Nil	<0.001	0.010	<0.001
202	<0.001	Nil	Nil	Nil	Nil	<0.001	0.008	Nil
202[2]	<0.001	Nil*	Nil**	Nil**	Nil*	<0.001*	0.009	<0.001*

[1] Exposure period of 360 hours.
[2] Sensitized 1 hour at 1200 F, air-cooled.
* Specimens lost metallic ring.
** Specimens embrittled by intergranular attack.

TABLE 6—Corrosion Rates Found in Organo-Nitrile Process (Test Period of 168 Hours)

Converter System	Corrosion Rate, ipy			
	316	304	202	201
	"As received" condition			
Top	0.008	0.163	0.091[1]	0.175
Bottom	0.004	0.142	Nil	0.127
Distillation	Nil	0.078[1]	0.002[1]	Nil
	Sensitized-one hour at 1200 F, air-cooled			
Top	0.015	0.072	0.268	0.219
Bottom	0.018	0.073	0.238	0.217
Distillation	0.000**	0.000	0.004*	0.000

[1] One out of two specimens in test remained completely passive.
* Significant loss of metallic ring.
** Slight loss of metallic ring.

deterioration as measured by this criterion while 304 and 201 alloy had not. Corrosion rates on specimens in the sensitized condition where attack was severe in the converter system are approximately three times as great for the 200 series alloys as for Type 304. Where the corrosion rates were rapid on the sensitized material, attack was by uniform loss of metal and there was no evidence of intergranular deterioration.

Other Environment

The four test environments just previously discussed summarize the results of the most recent tests conducted by this laboratory on the corrosion resistance of the chromium-nickel-manganese steels. In retrieving and collating the balance of the information that has accumulated in the authors' files for the past five years on the corrosion resistance of these materials, it appears advisable for the sake of brevity to categorize the corrosion data under the following seven environments:

(1) Organic aldehydes
(2) Organic acids
(3) Esters and esterification reactions
(4) Chloride exposures
(5) Sulfates
(6) Organo-nitrogen compounds
(7) Miscellaneous exposures

Pertinent information relative to the corrosion resistance of the 200 series compositions and the 18-8 steels in each of the seven categories is summarized in Tables 7 through 13.

The exposures are only generically defined in the tables of data, but the primary corrosion constituent is specified. Since interest here is directed only to a comparison of these new materials with the older 300 series stainless steels, the specific analysis of the exposure media is of no real interest. The question is not that of specific corrosion rates in a given medium, but what are the comparative values of resistance between the 200 and 300 series stainless steels.

A number of the exposures presented in the tables show "nil" corrosion rates for both the 200 and 300 stainless steels. Although this might be construed as providing no definite data, it does point out in most instances that the 200 series metals will not be attacked when the rate of attack on the 300 series is low or negligible.

Organic Aldehydes

Table 7 shows the results obtained by the exposure of the lower manganese, experimental heats of the alloys in organic aldehydes. Such exposures contain quantities of the corresponding organic acid. Note that the low manganese materials were as resistant as the conventional Type 304 alloy, although rates as high as 0.1 ipy were obtained.

Organic Acids

Acetic acid is a corrosive commonly encountered in industry today. Table 8 presents data comparing the six test alloys with the 300 series steels in exposure

TABLE 7—Corrosion of Chromium-Nickel-Manganese and 18-8 Chromium-Nickel Steels in Aldehyde Environment

Environment	Temp., Degrees C	Hours	Metal	Corrosion Rate, ipy Liquid	Vapor
Crude aldehyde distillation	60	480	304	0.008	0.093
			304[1]	.....	0.085
			GM-83	.....	0.065
		264	304	.....	0.021
		264	GM-82	0.005	0.027
Aldehyde containing 10 percent CO_2 with trace of formic acid	280	616	304	.....	<0.0001
			GM-83	.....	0.0001
	40	616	304	.....	0.0001
			GM-83	.....	<0.0001
Dialdehyde storage	30	4000	304	0.0004	.....
			GM-82	<0.0001	.....
	30	800	304	0.0001	.....
			GM-82	<0.0001	.....

[1] Sensitized 1 hour at 1200 F, air-cooled.

TABLE 8—Corrosion of Chromium-Nickel-Manganese and 18-8 Chromium-Nickel Steels in Organic Acid Environment

Environment	Temp., Degrees C	Hours	Metal	Corrosion Rate, ipy Liquid	Vapor
Acetic acid-acetic anhydride (Also See Table 4)	130—155	1115	316[1]	.....	0.011
			304[1]	0.005	0.058
			304	0.003	.....
			201	0.007	0.160
			201[1]	0.015	0.115
			202	0.006	0.080
			202[1]	0.024	0.091
			304	.....	0.013
			304[1]	.....	0.019
			201	.....	0.019
			201[1]	.....	0.032
			202	.....	0.020
	130—155	1115	202[1]	.....	0.034
	130	4800	304	0.0002[2]	0.006
			R-975	0.0005[2]	0.010
			R-976	0.0004[2]	0.017
	105	17,688	316	0.002	.....
			304	0.004	.....
			GM-82	0.003	.....
	117	2736	304L	0.071	.....
			GM-82	0.104	.....
	110	2736	304L	.....	0.048
			GM-83	.....	0.068
	100	2736	304	.....	Nil
			GM-82	.....	Nil
	30	4800	304	Nil	.....
			R-975	Nil	.....
			R-976	Nil	.....
Weak acetic acid	See Table 3				
Acetic acid-acetonitrile	103	94	304	0.004	.....
			GM-82	0.017	.....
	30	2130	304	<0.0001	.....
			GM-82	<0.0001	.....
Aldehyde oxidation	80	161	304	.....	0.001
			GM-83	.....	0.002
Acid-anhydride distillation	210	157	316	0.011	.....
			304	0.062	.....
			GM-83	0.029	.....
			R-975	0.023	.....
			R-976	0.026	.....
	245	50	316	0.014	.....
			304	0.033	.....
			GM-82	0.030	.....
	265	50	316	0.002	.....
			304	0.041	.....
			GM-82	0.041	.....
Unsaturated acid 20%–ether 75%–water 5%	30	184	304L	0.008	.....
			R-975	0.010	.....
			R-976	0.016	.....
Unsaturated acid 20% in organic medium	30	184	304L	<0.0001	.....
			R-975	<0.0001	.....
	100	184	304L	.....	0.100
			R-975	.....	0.118
Mixture containing 1% acetic acid in water	50	2880	304	0.002*	.....
			R-975	0.006*	.....
Resin preparation with acetic acid present	50	978	304	Nil	.....
			202	Nil	.....
		1512	304	Nil	.....
			202	Nil	.....
Unsaturated organic acid in water	50	696	304	Nil	.....
			GM-82	Nil	.....
Aryl acid, water, trace HCl	30	1296	316	0.0001	0.0005
			R-975	0.0007	0.0026
			R-976	0.0004	0.0022

[1] Sensitized-1 hour at 1200F, air-cooled.
[2] Test temperature 30C.
* Pitted.

to this acid as well as higher organic acids.

The molybdenum bearing stainless steels are the finest materials for this service, of course, but the Type 304 alloy may be used in exposures of moderate temperature. The 200 series metals in the annealed condition appear to have a corrosion resistance comparable to the 304 alloy in these media. The resistance of the 200 series steels improves and is fairly good in the higher organic acids. Also note that the 200 series steels were not attacked when the Type 304 alloy showed "nil" rates in the various exposures. However, one of the most significant aspects of the corrosion of the 200 series metals was the intergranular attack produced in two of the media. Although "nil" rates were obtained in the exposures, the 200 series steels were attacked to the point where the metallic ring of the coupons was lost, and subsequent examination showed extensive intergranular corrosion of sensitized specimens. This is more fully covered under the subject of the sensitization of these alloys.

Esters

From the corrosion standpoint, the data of Table 9 are primarily an exposure of metals in organic acids. A small amount of inorganic acid is usually present also.

The corrosion rates obtained by the exposure of metals in plant equipment producing organic esters followed the same pattern as that observed in the exposures in pure acid solutions. The 200 series steels were only slightly less corrosion-resistant than the 300 series. When the corrosive attack was mild, the 200 series compared favorably with the 300 series materials.

Chloride Exposures

The 200 series steels were indicated to have a resistance to chloride exposures similar to that of the 300 series steels. In general, the corrosion rates were the same, and the 200 series metals did not reveal any greater tendency to pit.

The last exposure shown in Table 10 involved the use of a heavy metal chloride brine containing ammonium chloride. Note that the 200 series metals were superior to other alloys under these conditions. The reasons for this greater chemical resistance are not apparent, but the results show the independent corrosion characteristics of a new alloy in conventional media.

Sulfate Exposures

A variety of exposures in both organic and inorganic sulfate solutions (Table 11) indicated that the 200 series steels were not sufficiently resistant to this type of ion attack. No truly significant difference in the rate of attack on the 200 and 300 series steels was observed, and more extensive testing would be required to determine the real characteristics of the 200 series steels in the media. A certain amount of risk is always involved in the use of any of the austenitic stainless steels in sulfate exposures, and consequently such results may be only of academic interest.

Organo-Nitrogen Compounds

The most interesting results obtained by the exposure of the 200 series steels in plant equipment are presented in Table 12. After tests were conducted in most types of organo-nitrogen compounds, it was concluded that the 200 series steels offer a chemical resistance to these media somewhere between that of Type 304 and Type 316 stainless steel.

In some instances, particularly those involving nitriles or conventional amine compounds, the superior resistance of the 200 series steels was significant. Such results are highly encouraging, and emphasize the fact that in the development of any new alloy categorical statements that it is "better" or "worse" than existing alloys may be misleading.

The most discouraging factor in the

TABLE 9—Corrosion of Chromium-Nickel-Manganese and 18-8 Chromium-Nickel Steels in Organic Ester Preparations

Environment	Temp., Degrees C	Hours	Metal	Corrosion Rate, ipy	
				Liquid	Vapor
Acetic acid, ester and alcohol	125	519	304	0.030	0.015
			R-975	0.042	0.019
	30	1345	304	Nil	.....
			R-975	Nil	.....
Organic acid, ester and alcohol	120	500	316	0.022	.....
			R-975	0.357	.....
			R-976	0.456	.....
	90	24	316	0.033	.....
			R-975	0.993	.....
			R-976	0.542	.....
	160	24	316	4.5	.....
			R-975	8.5	.....
			R-976	8.5	.....
	70	154	304	0.0007	.....
			R-975	0.0002	.....
			R-976	Nil	.....
Aryl acid-chloralcohol-ester	120	750	304	Nil	.....
			GM-83	Nil	.....

TABLE 10—Corrosion of Chromium-Nickel-Manganese and 18-8 Chromium-Nickel Steels in Chloride Exposures

Environment	Temp., Degrees C	Hours	Metal	Corrosion Rate, ipy	
				Liquid	Vapor
Ethyl and vinyl chloride solution	0	2528	304	Nil	.....
			R-975	Nil	.....
	30	1344	304	Nil	.....
			R-975	0.0001	.....
Distillation of an alkyl chloride	115	256	304	0.004	.....
			202	0.003	.....
	80	256	304	.....	0.001
			202	.....	0.001
Resin containing organic chlorides	150	360	304	0.001	0.001
			R-975	0.001	0.001
Aryl acid, alcohol, water, and hydrochloric acid	30	436	304	0.008	.....
			R-975	0.011	.....
			R-976	0.017	.....
Ether-hydrogen chloride solution (wet)	30	216	316	0.004	.....
			304	0.007	.....
			GM-83	0.003	.....
Ether-hydrogen chloride solution, (dry)	30	312	316	0.002	.....
			304	0.006	.....
			GM-83	0.005	.....
	100	67	316	0.118	.....
			304	0.297*	.....
			GM-83	0.687*	.....
	100	23	316	0.005	.....
			304	0.005	.....
			GM-83	0.006	.....
Carbon tetrachloride	150	48	316	0.001	.....
			201	0.001	.....
Chloroform	150	48	316	0.003	.....
			201	0.006	.....
Heavy metal chloride brine, aqueous 10% containing ammonium chloride	220	24	316	0.067	.....
			304	0.037	.....
			202	0.021	.....
	220	84	304	0.022	0.001
			202	0.005	0.003
	102	168	304	0.0001	.....
			202	0.0002	.....

* Pitted.

use of the 200 series alloy is again illustrated by the intergranular attack which occurred on specimens exposed in two of the amine media. In one organic nitrile preparation and in an aromatic diamine preparation a loss in the mechanical properties of the alloys was experienced (Table 5). The first of these exposures showed a considerable increase in the rate of attack on the sensitized specimens, but the last exposures produced no significant difference by this method of evaluation.

It is interesting to note in these data that in the exposures at higher temperatures or in those media where the corrosion rates were most severe, these materials provided some excellent results. Although they cannot compare with the molybdenum bearing stainless steels in such service, the 200 series steels may find useful service in such process equipment if the problem of welding and resulting sensitization can be mitigated.

TABLE 11—Corrosion of Chromium-Nickel-Manganese and 18-8 Chromium-Nickel Steels in Sulfate Exposures

Environment	Temp., Degrees C	Hours	Metal	Corrosion Rate, ipy	
				Liquid	Vapor
Alkyl sulfate distillation	50	288	304	0.006	0.006
			R-975	0.011	0.011
			R-976	0.005	0.005
	70	288	304	Nil	Nil
			R-975	0.0002	0.0002
			R-976	0.0001	0.0001
	60	153	304	Nil	0.004
			GM-82	0.0003	0.001
	85	153	304	0.015	0.003
			GM-82	0.014	0.004
Alkyl acid sulfate reaction	90	96	304	0.015	0.021[1]
			R-975	0.018	0.004
			R-976	0.033	0.001
Phenols with 5% H_2SO_4 present	205	1080	304	.....	0.0005
			GM-82	.....	0.0009
Alcohols with 1% sulfuric acid	40	1176	304	0.011	.....
			R-975	0.070	.....
			R-976	0.080	.....

[1] Sensitized 1 hour at 1200 F, air-cooled.

Miscellaneous Exposures

Table 13 provides information on the exposure of the alloys in a variety of chemicals. In general, the 200 series alloys revealed excellent chemical resistance and were as satisfactory in the annealed condition as the Type 304 alloy for general chemical service.

Exposures in phosphoric acid media produced low corrosion rates and thus no significant difference could be determined between the various alloys. In phenols at high temperatures, the 200 type alloy was highly resistant. Pitting of the metal resulted only when pitting of the Type 304 metal also occurred. These data, combined with the excellent results obtained in the amine exposures, suggest that the 200 series stainless steels may have an inherent resistance to organic bases exceeded only by the molybdenum bearing stainless steels and the Hastelloy alloys. There is some indication in Table 13 that the 200 series may not have resistance to the halogen compounds comparable to that of the Type 304 alloy.

TABLE 12—Corrosion of Chromium-Nickel-Manganese and 18-8 Chromium-Nickel Steels in Organo-Nitrogen Compounds

Environment	Temp., Degrees C	Hours	Metal	Corrosion Rate, ipy	
				Liquid	Vapor
Aqueous solution of hydrogen cyanide, ammonium sulfate and excess of H_2SO_4	110	24	304	0.001	0.001
			202	0.001	0.001
	110	552	304	.....	0.148
			201	.....	0.0001
	70	26	304	0.0005	.....
			202	Nil	.....
	55	671	304	.....	0.013
			GM-83	.....	0.005
	55	2016	304	.....	0.011
			R-975	.....	0.031*
			R-976	.....	Nil
	60	671	304	.....	0.013
			GM-83	.....	0.010
	65	1464	304	.....	0.105
			R-975	.....	0.206
			R-976	.....	0.192
Aqueous hydrogen cyanide, 20%	55	3144	304	0.005	.....
			GM-83	0.004	.....
Aqueous hydrogen cyanide, 35%	70	2016	304	Nil	.....
			GM-83	Nil	.....
			R-975	Nil	.....
			R-976	Nil	.....
Organo-nitrile converter	350	40	304	.....	0.0020
			GM-82	.....	0.0016
Organo-nitrile converter	80	68	304	Nil	.....
			GM-82	0.0003	.....
Organo-nitrile converter product	80	68	304	Nil	.....
			GM-82	0.0003	.....
Crude organo-nitrile distillation	90	408	304	0.014	0.014
			GM-83	0.012	0.014
	75	432	304	0.0025	0.003
			GM-83	0.0036	0.004
Organo-nitrile preparation	See Table 6				

Environment	Temp., Degrees C	Hours	Metal	Corrosion Rate, ipy	
				Liquid	Vapor
Agricultural chemical preparation containing amines and sulfides	165	436	304	0.268	.....
			GM-83	0.370	.....
	165	528	316	0.015	.....
			GM-82	0.274	.....
Alkyl diamines	110	744	304	Nil	Nil
			R-975	Nil	Nil
			R-976	Nil	Nil
Alkyl pyridine preparation	150	1056	304	.....	0.022
			R-975	.....	0.001
	195	1056	304	.....	0.003
			R-975	.....	0.001
	200	1056	304	.....	0.013
			R-975	.....	0.008
	140	1056	304	.....	0.056
			R-975	.....	0.055
	100	456	304	.....	0.004
			R-975	.....	0.002
	150	850	304	.....	0.107
			R-975	.....	0.051
	190	850	304	.....	0.084
			R-975	.....	0.004
Pyridine-aldehyde reaction	240	24	304	.....	0.0007
			GM-82	.....	0.0013
Aromatic diamine preparation	40	360	347	0.0002	.....
			202	0.0001	.....
			202[1]	0.0003	.....
	135	360	347	.....	0.009
			202	.....	0.007
			202[1]	.....	0.009
	280	360	347	.....	0.0001
			202	.....	0.0001
			202[1]	.....	0.0001
Triethanolamine	140	168	GM-82	0.0002	.....
Diethanolamine	140	168	GM-82	0.0002	.....
Monoethanolamine	140	168	GM-82	0.0002	.....

[1] Sensitized 1 hour at 1200F, air-cooled.
* Pitted.

TABLE 13—Corrosion of Chromium-Nickel-Manganese and 18-8 Chromium-Nickel Steels in Miscellaneous Exposures

Environment	Temp., Degrees C	Hours	Metal	Corrosion Rate, ipy: Liquid	Corrosion Rate, ipy: Vapor
Hydrocarbon, H_3PO_4, CO_2 and water, pH-2	150	336	304	Nil	Nil
			R-975	Nil	Nil
			R-976	Nil	Nil
Hydroxy alkane with 0.006 percent H_3PO_4	130	48	304	0.0002	.....
			R-975	0.0002	.....
			R-976	0.0001	.....
Phenols, aryl bases, oils, trace of sulfur	300	1016	304	.....	0.0001
			GM-83	.....	<0.0001
	300	1016	304	.....	0.0211
			GM-83	.....	0.0005
	300	1016	304	.....	0.070*
			GM-83	.....	0.031*
	200	2415	304	.....	Nil
			R-975	.....	Nil
			R-976	.....	Nil
	320	2415	304	.....	0.0002
			R-975	.....	Nil
			R-976	.....	Nil
	340	2415	304	.....	0.0003
			R-975	.....	Nil
			R-976	.....	0.0007
	170	1224	304	.....	Nil
			GM-82	.....	Nil
Alkyl phenol preparation	30—200	867	304	.....	Nil
			R-975	.....	Nil
			R-976	.....	Nil
2.5% chlorine in moist air	30	95	316	.....	0.005*
			304L	.....	0.007*
			202	.....	0.003*
			202[1]	.....	0.004*
35% aqueous boric acid	100	600	304	Nil	.....
			R-975	Nil	.....
			R-976	Nil	.....
Miscellaneous alcohols, chlorides, amines, and ester distillations	.....	944	304	0.0006	0.0008
	.....		R-976	0.0015	0.0020
Ketone distillation	140	163	304	0.013	0.003
			304[1]	0.035	0.010
			R-975	0.036	0.009
			R-976	0.028	0.005
	140	600	304	0.002	0.003
			GM-82	0.003	0.005
2% agricultural chemical (aerated)	30	312	304	Nil	.....
			GM-83	Nil	.....
Alcohol, glycol, boron trifluoride solution	170	1656	304	0.049	.....
			R-976	0.083	.....
Perfluoroalkane with peroxide present	0	1484	316	0.0002	.....
			304	0.0002	.....
			R-975	0.0002	.....
			R-976	0.0003	.....
Perfluoroalkane in Freon	0	160	304	0.001	.....
			R-975	0.001	.....
			R-976	0.001	.....

[1] Sensitized 1 hour at 1200F, air-cooled.
* Pitted.

Summary and Conclusions

The results of exploratory field test exposures in various chemical plant processes have indicated that the present compositions of the chromium-nickel-manganese steels designated as AISI types 201 and 202 are about equally corrosion resistant in most chemical environments tested.

These steels in the annealed condition, it was indicated, may be used in a number of corrosive environments in the chemical manufacturing industry where Type 304 is being utilized in large quantities. Some examples of services where these steels may be used as substitutes for Type 304 are: organic acid exposures at moderate temperatures, aldehyde service, phenol exposures, and in the majority of organic product handling and storage at ambient temperatures. Where organo-nitrogen compounds or conventional amines are the major corrosive constituents encountered, the 200 series steels offer somewhat better corrosion resistance than Type 304.

Environments in which the 200 series steels do not appear to be the equivalent of Type 304 are organic acid-anhydride solutions at accelerated temperatures and certain esterification reactions. In the presence of halogen compounds and possibly in sulfate media, the chromium-nickel-manganese steels were less corrosion resistant than Type 304.

After moderate sensitizing heat treatments of 1 hour at 1200 F the 200 series alloys were more susceptible to intergranular corrosion than Type 304 during exposure under operating conditions in some chemical manufacturing processes. As previously pointed out, results of the field tests do not corroborate the results obtained and reported by others on sensitized specimens exposed in the standardized evaluation tests.

Inasmuch as the 200 series alloys are not readily amenable to the addition of stabilizing elements, the variable results obtained with respect to susceptibility to intergranular attack points to the conclusion that extra-low-carbon grades of the 200 series alloys should be more fully investigated for replacement of Type 304 in the more corrosive chemical services. These low-carbon heats should be made available for testing and should be included in all of the testing programs designed to extend the use of these materials as a means of conserving nickel.

Acknowledgment

This investigation was undertaken in co-operation with the Union Carbide Metals Company. The suggestions, contributions, and assistance of W. O. Binder, Assistant Director of the Development Department of the Union Carbide Metals Company, who supplied the experimental heats of steel reported in these tests, are gratefully acknowledged.

The authors are grateful to R. Franks and G. A. Sands for their continued interest and support of the test program, and for their kindness in reviewing the manuscript.

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DISCUSSION

Comments by H. Ebling, A. O. Smith Corp., Milwaukee, Wisconsin:

The authors of this paper have presented a great deal of information concerning the relative corrosion resistance of the new "200" series of stainless steels and the established "300" series in various chemical processes. From this investigation it was revealed that in a number of service applications, the AISI types 201 and 202 could be substituted for Type 304. This conclusion was based on the results of exposed samples of the material in the as received condition

and in a moderate sensitizing heat treatment of 1200 F for one hour.

This type investigation certainly screens out processes which are too severe for the innate corrosion properties of a material. However, even after the material is found to have adequate corrosion resistance to a process, there are a number of other factors which still must be considered. Unfortunately it is difficult to fabricate a useful article from the annealed condition and still expect the material to have the same corrosion resistant properties. Some of the questions that might be encountered by a fabricator are as follows:

1. Is contact or crevice corrosion a problem?
2. What are the probabilities of galvanic corrosion?
3. Will the material be satisfactory after hot forming, stress relieving, or welding?
4. After fabrication must the article be chemically cleaned to prevent possible pitting?
5. Is the material satisfactory after cold forming, bending, or other conditions where the material may be highly stressed?

The answers to some of the above questions are difficult and have been obtained on well known materials through experience over many years. In obtaining these answers I suspect that some fabricators and users of the materials paid very dearly for some of their experience. The authors have attempted to answer some of these questions inasmuch as some samples were sensitized. This suggests that the authors were aware that the as received condition of the materials might be changed in fabrication. This could have been carried out a little further to include a weld deposit on the exposed specimens. In doing so, the specimen would have the actual heat affected zone adjacent to the weld which in many cases cannot be simulated by some sensitizing heat treatment. It would have introduced another metal; as far as known there are no electrodes of the 200 series type, which might give some insight as to possible galvanic corrosion.

The authors observed that in certain processes there was a possibility of stress corrosion occurring. This is especially interesting since the specimens were prepared to minimize shear strain. Pitting attack was observed and noted in some instances.

Laboratory corrosion tests made on the "200" series of stainless steel generally show lower corrosion resistance than the "300" series in so far as the nitric acid test is concerned. I believe it is generally known that the "200" series will sensitize more readily from stress relieving, welding, etc. than the "300" series. It appears that the material is more susceptible to stress corrosion than the "300" series. With the evidence on hand, it is imperative that some caution be observed before any wholesale substituting of "200" series steels for the "300" series steels is done. Otherwise we will be again gathering very expensive experience.

Reply by A. C. Hamstead and L. S. VanDelinder

The present evaluation of a new material was made to obtain data on the basic alloy and to point out certain corrosive chemical environments wherein further investigation of the 200 series steels may be warranted, as well as environments wherein such studies may not be warranted. Such questions as those regarding fabrication difficulties which might be experienced are not pertinent to our investigation on general corrosion resistance of the material. We would leave the problem of the hot working properties and welding techniques to those directly concerned with the problem. It is difficult to believe that the 200 series austenitic steels would differ significantly from the 300 series in such characteristics as crevice corrosion or in weakness to stress cracking in certain environments. It is difficult to believe that electrochemical potentials of this material would be significantly different from those of the 300 series materials.

As for the testing of weld specimens, we would like to add to what has already been said that we are reticent to accept an evaluation of such coupons as a definitive answer to the problems encountered by welding or stress relieving of an austenitic alloy. If carbide precipitation is known to occur in the sensitizing range, stabilization or annealing of the alloy is mandatory for chemical service where intergranular attack will occur. Severe sensitizing heat treatments provide uniform data concerning the propensity of the alloy to corrode excessively in such environments. Variations in welding technique used on a particular analysis of material may preclude such corrosive attack but we do not care to specify a carbide precipitating alloy on the basis of such a possibility.

Problems of fabrication yet to be answered point out the need for a wider interest by all parties concerned and greater action for the development of this alloy. The time may come when this material is again of prime interest and it is not wise to wait until sufficient time is not available to properly evaluate the material.

Comments by W. G. Renshaw, Allegheny Ludlum Steel Corp., Brackenridge, Pa.:

The authors have presented results of some very thorough and valuable investigation on performance of Cr-Ni-Mn steels in actual service in chemical processes. Although considerable laboratory data have become available, it is only through service test experience that best evaluation of materials can be made.

It is encouraging to note that in general the 200 series in the annealed condition may be used in a number of corrosive environments in the chemical manufacturing industry where Type 304 is presently being used in large quantities. It is especially significant to note superior behavior shown by the Cr-Ni-Mn steels in organo-nitrogen compounds or conventional amines.

Comparing the authors' plant experience with these steels after sensitizing, to laboratory evaluation tests serves to illustrate forcefully that no direct relationship exists. While standardized tests offer considerable worth for control work, the only useful evaluation is obtained under actual service conditions.

The only unfortunate circumstance, as they mention, is that 204L material had not received more testing. Since the precipitate at the grain boundaries in Type 204L is discontinuous it would have been interesting to learn how this might have performed in the same environments. The carbon contents of the Cr-Ni-Mn steels in tests where sensitization was employed were generally on the high side (.06 percent and .09 percent) except for a few isolated tests with Type 204L. Only in Tables 3, 4 and 6 was Type 304 included in the sensitized condition in plant media and in one of these instances even Type 316 showed greater intergranular susceptibility than Type 304.

While welding of materials involves heating within the carbide precipitation range it usually does not involve holding at temperatures as long as one hour. For this reason it would seem that heating one hour at 1200 F is not realistic except for stabilized grades or .03 percent maximum carbon grades. Actual welded specimens of Types 201, 202 and 304 in the media shown probably would be more representative.

Reply by A. C. Hamstead and L. S. VanDelinder

It is true that data on actual service performance are most convincing and trustworthy. But before this type of information will be forthcoming on the 200 series steels from the chemical industry, one of the problems confronting the introduction and use of a material for "new" applications will have to be overcome by one means or another. From the time of adoption of these materials as standards by AISI until the conclusion of our work, inquiries on these materials for chemical process equipment has disclosed that: (1) The materials were not readily available, (2) Many fabricators are not willing to quote on chemical process equipment fabricated of these materials and, (3) There is very little price advantage of these materials over Type 304. It is possible that this situation may have improved a little recently. However until a definite improvement is noted in availability, and willingness to fabricate chemical equipment using these materials, 200 series steels cannot be expected to gain much use in the chemical industries until another national emergency arises.

The sensitizing treatment used for the test specimens, we agree, was not a realistic treatment for simulating conditions in welded construction for regular grades. Nevertheless, this treatment is the one most generally used and prescribed for determining relative susceptibility of a material to intergranular attack, and would reasonably well simulate conditions encountered in special heat treatments such as stress relieving, etc. This treatment was about the same as the one described in the ASTM procedure for

testing susceptibility of the stabilized and low-carbon grades to intergranular attack. It is difficult to determine what a realistic sensitizing treatment would be for simulating welding conditions since so much would depend upon plate thickness, speed of welding, and procedures. Actually, welded specimens themselves will not provide the entire answer to susceptibility of chemical equipment fabricated of unstabilized grades to intergranular attack unless the specimens are welded of the same materials using the same techniques and procedures as prescribed for the particular vessel. The concern over testing specimens of various degrees of sensitization, or welded specimens, becomes less important when it is realized, as pointed out in the previous discussion to the comments of Mr. Ebling, that annealing after fabrication (welding) would be mandatory in many cases. This would be true for all regular grades of steel in which carbide precipitation is known to occur at the grain boundaries whenever these steels are specified for corrosive services in which intergranular corrosion may possibly occur.

Comments by M. A. Scheil, A. O. Smith Corp., Milwaukee, Wisconsin:

After reviewing the well written paper of the authors we note that both T-201 or T-202 carry a carbon specification of 0.15 percent maximum, yet the testing is carried out on alloys under 0.10 percent carbon. If this wide range of carbon is necessary for the manufacturer to protect himself and has the sanction of AISI, then the authors are amiss in not reporting one heat that borders on the maximum of the range to include in their series of tests.

The paper is not clear on what grade of surface was used in testing. Is this No. 2, 2B or something else?

On page 67 reference to loss of metallic ring, in conjunction with satisfactory bend tests contradicts the ASTM where either loss of metallic ring or failure in bend rejects the material. How then by their test does the user know he has suffered intergranular attack?

For the sake of clarity I would suggest that the authors prepare polished micro sections of their bend tests and examine same for intergranular cracks.

It is difficult for the writer to get very excited over this substitute alloy when the authors do not include tests of the material in the weld fabricated condition. We would like to see a comparison of the corrosion characteristics of "weldments" between the 200 and 300 series stainless steels in a number of service applications on which the authors report.

Reply by A. C. Hamstead and L. S. VanDelinder:

The surface finish of the commercial heats of 201 and 202 steel was designated as 2B on the invoice from the supplier. The experimental heats were supplied as strips that had been formed from small ingots by hot working at an initial temperature of 1150 to 1175 C, and air-cooled, then descaled. The surface finish of these specimens was dull with a roughness approximating that of a light sandblast. Other than the cleaning procedure described under "Methods," the original surface finish of the specimens was not disturbed for the tests.

The sensitized 200 series specimens that were reported to have lost their metallic ring yet retained sufficient ductility to be bent through 180 degrees without cracking were, upon metallographic examination, found to be intergranularly corroded to an approximate depth of 2 to 3 grains. Re-examination of these bend specimens under the microscope at low-power in accord with ASTM, A393-55T, Specifications Test Procedure disclosed shallow fissuring across the face of the bends. The impression gained from the report that these bends would qualify satisfactorily under the ASTM procedure should be corrected. It should be explained that intergranular corrosion was first suspected by loss of metallic ring. The type of attack and extent was confirmed by metallographic examination and the relative degree indicated in footnotes to the table of results. When loss of metallic ring is noted in the tables, it may therefore be concluded that the specimens have suffered intergranular attack and would not necessarily pass the ASTM bend test for susceptibility to intergranular attack. However, we should like to add with reference to the comment of Mr. Scheil that, "either loss of metallic ring, or failure in bend rejects the material," that there is no mention in the present ASTM procedures of "loss of metallic ring" as a criterion for judging intergranular attack. So that even though one were to report a perceptible loss of metallic ring along with a satisfactory bend test, there would be no contradiction within the ASTM procedures.

The reason that welded specimens were not used in our tests is that at the start of this investigation (1952) we were primarily interested in comparing the general corrosion resistance of these "new" or "proposed compositions" with that of the 300 series steels for corrosive chemical service. These new compositions had not yet become AISI standards and we did not feel that a comprehensive, expensive investigation could be justified at this time on non-standard materials. In addition, at that time we did not have the necessary information on techniques for welding these materials. By the time these materials became standardized by AISI in 1955 and our commercial heats were available for testing, considerable information had been developed and published by others on the welding characteristics of these steels. Franks, Binder and Thompson had demonstrated that these steels "possessed adequate weldability for all types of practical applications." Renshaw, the authors mentioned, and Binder, Thompson and Bishop had determined that these steels, like the chromium-nickel steels were subject to intergranular corrosion when heated within certain temperature ranges (1000 to 1600 F). Recognizing that these materials were susceptible to intergranular corrosion, we felt that comparative data on sensitized specimens would be sufficient for purposes of our investigation.

The usefulness and convenience of testing welded specimens in preference to sensitized specimens for evaluating susceptibility of a material to intergranular attack in an environment is controversial. We would agree, however, that welded specimens should be tested for evaluating or qualifying welding procedures or special heat treatments on unstabilized grades prescribed for welded construction for specific chemical services.

Any discussions of this article not published above will appear in the June, 1959 issue

Control of Internal Corrosion of Petroleum Products Pipelines With Oil Soluble Inhibitors*

By M. R. BARUSCH, L. G. HASKELL and R. L. PIEHL

Abstract

This article summarizes the corrosion inhibitor program of a products pipeline system since its initial operation eight years ago. Initially alkaline sodium nitrite solutions were utilized for corrosion protection. This material was an effective corrosion inhibitor, but its use resulted in the production of large quantities of rust and scale, and in addition contributed to water cloud problems. Use of an oil soluble corrosion inhibitor minimized these problems and resulted in improved protection of the pipeline. During the past three years an average internal corrosion rate of only 0.025 mil per year was observed, in spite of the fact that prolonged periods occurred when no inhibited product contacted sections of the pipe.

A mechanism explaining the behavior of oil soluble corrosion inhibitors in a pipeline is presented. This theory accounts for the outstanding effectiveness of such materials and explains why they protect the metal during the prolonged periods when uninhibited stocks are present. The use of more than one oil soluble corrosion inhibitor in products transported through a pipeline causes mixtures of inhibitor molecules to be adsorbed on the surface of the pipe. One inhibitor in contact with the pipe tends to displace another inhibitor previously adsorbed on the surface. This results in some interchange of the corrosion inhibitors in the products transported. The degree of protection realized from the use of several corrosion inhibitors in a pipeline system is discussed. 5.8.2

Introduction

THE PRODUCTS pipeline system of the Salt Lake Pipe Line Company extends from Salt Lake City, Utah, to Spokane, Washington, a distance of 706 pipeline miles. In addition, there are 435 miles of double and loop line. The major portion of the trunk line is 8-inch, and the balance 6-inch pipe as shown on the general route map in Figure 1.

The system has grown from a single 8-inch and 6-inch line from Salt Lake City to Pasco, Washington, which was put in service in 1950 with an initial capacity of 16,000 BPD, to its present size with a capacity of 56,000 BPD. This system carries or has carried aviation gasoline, jet fuel, premium and housebrand motor gasolines, alkylate, stove oil, furnace oil, and Diesel fuel. Nine separate shippers use the system for transporting their petroleum products into the Northwest. More than 60 different grades and brands of products have been carried in separate batches during a single year. Most of the products enter the system at the Salt Lake Station, but a substantial quantity, transported by river barges from Pacific Coast refineries, is introduced into the system at Pasco, Washington.

In the earliest planning of the pipeline operations, it was recognized that internal corrosion could be an important problem. The experience of many other products pipeline systems indicated that large quantities of mill scale, rust, and foreign material could be expected for about a year before any system of corrosion inhibition could be depended upon to keep the internal surface of the pipe bright and smooth. Later in this paper, it is shown that this troublesome period can be avoided through use of oil soluble corrosion inhibitors.

The need for a means of protecting a products pipeline from internal corrosion arises because petroleum products carry water in solution. The amount of water dissolved in a product is dependent upon the temperature. At 110 F, a gasoline saturated with water contains about 0.05 percent by weight dissolved water.[1] This water, which is the primary cause of the internal corrosion of pipelines, is neither "free" water which can be settled, filtered, or adsorbed, nor water held in emulsified suspension. The water held in solution in products which are received by the pipeline shortly after manufacture, without benefit of standing in shippers' storage tanks long enough to cool, precipitates in the pipeline. The gasoline introduced into the pipeline may be as warm as 110 F and the gas oils may be 140 F, whereas normal pipeline temperatures range from 40 F to 75 F. The reduced solubility of water in the product at the lower temperatures results in precipitation of about 80 percent of the dissolved water from a product introduced into the pipeline at 110 F.[1]

MAURICE R. BARUSCH—Group Supervisor, California Research Corp., Richmond, California. He has AB and PhD degrees in chemistry from Stanford University. He is author and co-author of papers on subjects related to hydrocarbon combustion and has many patents on engine fuel additives.

L. G. HASKELL—Chief engineer, Salt Lake Pipe Line Co., Salt Lake City, Utah. With a BA in civil engineering, Stanford University, 1921, his entire professional experience has been with Standard Oil Company of California and its subsidiaries. His work includes design, construction and operation of oil pipe line systems. He is chairman of Salt Lake Section NACE.

ROBERT L. PIEHL—Materials Engineer, Standard Oil Co. of Cal., Richmond, California. He has a BS in chemical engineering, 1953, University of Wisconsin and since then has been concerned with corrosion in petrochemical and petroleum industries. He served as chairman of the program coordination committee for the 1958 NACE 14th Annual Conference.

Methods for Controlling Corrosion

There are two basic means of controlling internal corrosion of a pipeline. One, used by some operators, is to dehydrate all of the products prior to their introduction into the system. This is usually accomplished with a desiccant such as activated alumina. The spent desiccant may be regenerated by heating. The alternate method counteracts the corrosive effect of water in the products by the use of an adequate corrosion inhibitor. The Salt Lake Pipe Line Company elected to use the latter method because of economic considerations.

Corrosion inhibitors for pipeline use may be classified as those which are introduced in aqueous solution and those which are added directly to the petroleum products. Both have been used in the Salt Lake Pipe Line Company's system. When the first segment of the pipeline was ready for operation, California Research Corporation was completing the development of an oil soluble inhibitor to protect pipelines. However, this development was not then available to the Salt Lake Pipe Line Company. As a result of this situation and because of the opinions and experience of many other operators of pipelines that all of the mill scale must be removed from the internal surface of the new pipe, a water solution of sodium nitrite with a pH of 8.0-9.5 was used as an inhibitor during the early operations of the system. The use of such an alkaline sodium nitrite solution in pipelines was described by Smith, Curry, and Rush.[2]

The usual practice in using sodium nitrite as an inhibitor is to inject a suffi-

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cient quantity of the sodium nitrite and caustic soda to maintain 1 to 2 percent sodium nitrite and a pH between 8.0 and 9.5 in the water leaving the line. It is generally considered that the pipeline is completely protected if the effluent water meets these requirements.

Starting in June 1950, the inhibitor was injected into all of the product batches at the numerous pump and delivery stations along the line. With the advent of cool weather in the fall, a water cloud problem in the gas oils developed, and the injection was restricted to motor gasolines. This problem became so serious that the use of sodium nitrite was discontinued on an experimental basis, and the cloud problem improved. Renewal of sodium nitrite injection resulted again in the occurrence of a serious water cloud problem.

The operations of the pipeline with sodium nitrite as a corrosion inhibitor required frequent running of scrapers for the removal of scale and corrosion products. No permanent records were maintained of scraper runs and the amount of material that they removed. However, it is recalled that scrapers were run about once per week, daily in some sections of the line, and sometimes at longer than weekly intervals. The scrapers were always run in gasolines because the scale and corrosion products settle out more readily from gasolines than from oils.

A typical routine scraper run resulted in the removal of ten to fifteen barrels of rusty mill scale from each 40-50 mile section of pipeline. Additional rust was drained with contaminated product from piping and strainers into sump tanks, and finer material was suspended in products delivered to shippers' tanks. Some of the material was so fine that it imparted a color to products and was extremely difficult to remove by settling. On occasion, as much as 50 barrels of rust and scale was removed by a scraper from a section of the pipeline. Even after one year of operation with sodium nitrite inhibitor, there was little decrease in the quantities of rust being removed by the frequent passage of scrapers. This experience is similar to that reported by DeVerter and Jasek[3] on the use of a water soluble inhibitor in a products pipeline system.

All scrapers run as a part of pipeline operations are of the brush type which thoroughly scratch the entire internal surface of the pipe with tough, strong steel bristles. Figures 2 and 3 shows examples of such typical scrapers. The "cups" of the scraper body, fitting tightly in the pipe, enable the product stream being pumped to force the scraper along, while the wire brushes thoroughly loosen and remove any rust scale that may have formed in the pipe. Such scrapers are ingenious mechanisms and have been designed in recent years to fit two adjacent sizes of pipe. For example, the section of pipeline from Baker to Pasco (refer to Figure 1) is 8-inch about half the way, then 6-inch the remainder, with a short swage or taper joining the two sizes. The "double diameter" scraper fits the 8-inch pipe well and has collapsible vanes that enable it also to fit the reduced diameter of 6-inch pipe.

Corrosion Measurements

It was recognized that some control method should be used for determining the effectiveness of the corrosion inhibitor program. A roughness factor, used by hydraulic engineers, known as the "C" factor serves to determine the effects of corrosion and the benefits of inhibition. Such "C" factors can be conveniently calculated from the hydraulics characteristics when batches of a single product are large enough to fill the line between reference locations. Changes in the "C" factor then establish the changes in roughness of the internal surfaces. Such a calculation becomes laborious and the results somewhat nebulous when it is determined under a wide variation of operating conditions, temperatures, pressures, product gravities, viscosities, and flow rates. "C" factors so obtained are at best only a comparative measure rather than a quantitative measurement of roughness. Such calculations do not readily determine how much of the material is continuously eroded from the internal surface of the pipe.

Use of Test Coupons

Exposure of test coupons is another means commonly used for determining the rate of corrosion of the internal surface of the pipe. This plan was adopted because of its simplicity and accuracy by the Salt Lake Pipe Line Company. The coupons must be of a composition similar to that of the pipe. For convenience, they should be placed where they are reasonably accessible without shutting down the system for installation and

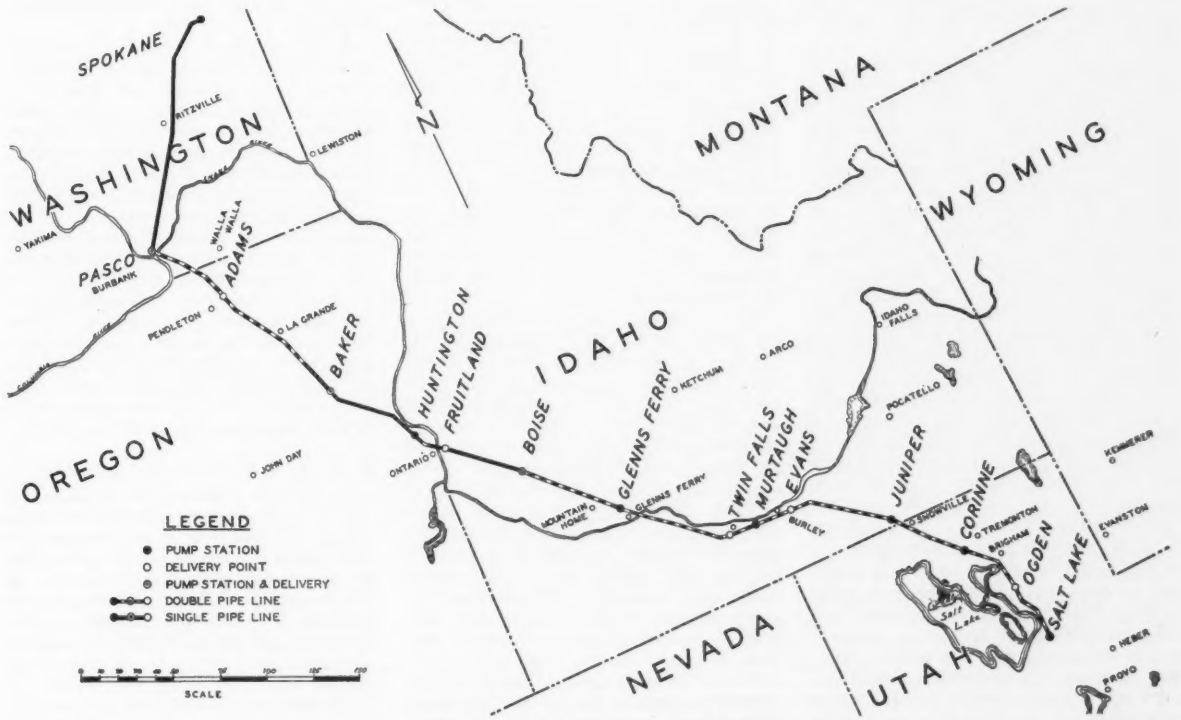

Figure 1—Route of Salt Lake-Spokane products pipeline.

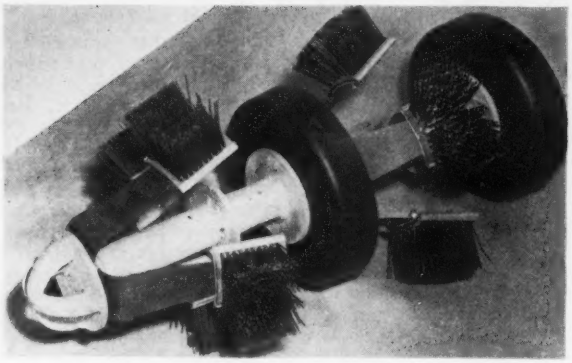

Figure 2—Eight-inch type WC-11 scraper with two rubber cups. Photo courtesy of T. D. Williamson, Inc., Tulsa, Okla.

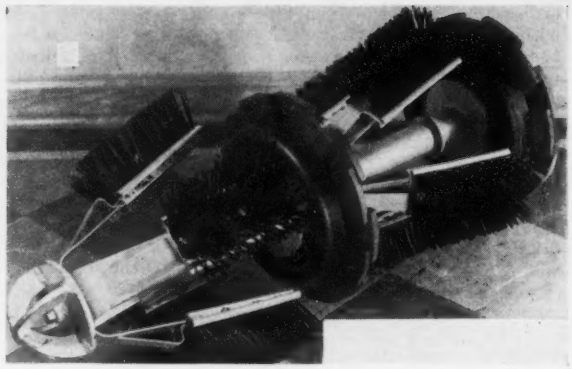

Figure 3—six-inch by eight-inch type WCK-11D duo-disc scraper for running in six-inch and eight-inch pipe. Photo courtesy of T. D. Williamson, Inc., Tulsa, Okla.

removal. They should be so placed that a representative product stream passes by them at all times. Furthermore, they should be placed in locations where the velocity and temperature of the product stream are typical of those occurring in the pipeline. Accordingly, coupons were placed in the streams received at Boise, Pasco, and Spokane, At Pasco, some new products were received into the system from shippers' terminal tanks supplied from river barges. These products sometimes contained water in quantities not found in the products transported solely by pipeline. For this reason, test coupons also were placed in the piping which received product streams from these outside sources. As a further check on the conditions in the system, test coupons were placed in the outgoing stream from Pasco Station. A schematic flow diagram of the entire products pipeline system and the placement of the test coupons is shown in Figure 4.

Test coupons were prepared from 16-gage mild steel (SAE 1020) sheet, 1¾ inches wide and 4½ inches long with a 7/16-inch hole near one end. All surfaces were given a uniform finish on a belt sander using a 150 grit paper, after which the specimens were degreased with acetone and weighed. Because of the time and distance involved in shipping the specimens to points of installation in the pipeline, they were coated with a light, noninhibited oil to prevent surface rusting in transit. After being removed from the pipeline, the test specimens were again coated with the light oil and returned for evaluation. Exposed coupons, when received, were cleaned with a mild abrasive soap and water, dried with acetone, and reweighed. In view of the length of specimen exposure and the fact that the specimens in most cases showed only traces of surface rust, the cleaning procedure caused no significant error in corrosion rate determinations.

The initial installation of corrosion test coupons in the pipeline was made in July 1951. After experimenting with different periods of specimen exposure, it was concluded that the most satisfactory program would provide control results every three months from coupons exposed during the preceding six months. Corrosion rates obtained from specimens exposed during the early period of operation of the pipeline together with a brief description of the corrosion inhibitor program are summarized in Table 1.

During the year 1951, it was possible to obtain a measurement of the corrosion rate in piping at a location upstream from the point of inhibitor injection. At this time, a corrosion rate of 1.2 mils per year was obtained. The present method of pipeline operations no longer permits measurements of corrosion in the presence of uninhibited products, but such measurements currently being made in another products pipeline operated in a similar manner show corrosion rates as high as 3.5 mils per year and an average of 1.5 mils per year during the past two years.

Use of Oil Soluble Inhibitor

Use of the sodium nitrite inhibitor reduced the corrosion rate to an average of 0.15 mil per year. Despite an approximately tenfold reduction in corrosion rate, the water cloud problem discussed previously forced discontinuing the injection of sodium nitrite in the fall of 1951. At this time, the Salt Lake Pipe Line Company started using Oronite R and T corrosion inhibitor, which is of the oil soluble type. This inhibitor was injected into motor gasolines at the initial station at Salt Lake. Motor gasolines comprise about 60 percent of the products transported.

Oil soluble corrosion inhibitors have an advantage over the water soluble inhibitors because the latter require frequent injection stations along the pipeline. The use of the oil soluble inhibitor reduced the corrosion rate to an average of 0.04 mil per year, greatly improved the water cloud problem, and nearly eliminated the need for the running of scrapers. Since the introduction of oil soluble inhibitors, it has been found that quarterly scraper runs bring in little or no rust. The effectiveness of oil soluble inhibitors is demonstrated by the fact that scrapers need be run only for the purpose of confirming the very low corrosion rates shown by the test coupons.

Within recent years, it has become a relatively common practice for petroleum refiners to incorporate oil soluble corrosion inhibitors in their products. Salt Lake Pipe Line Company, as a common carrier, has no control over or full knowledge of the types of additives introduced at the refineries. While the primary function of such additives is the improvement of the product quality, they also may be of benefit in controlling corrosion in the pipeline. Prior to 1953, it is believed that none of the products transported in the pipeline contained any corrosion preventive other than the Oronite R and T inhibitor. The low corrosion rate of 0.04 mil per year that was realized was, therefore, due solely to the use of this inhibitor.

In early 1953, a second corrosion inhibitor was introduced into the pipeline system by virtue of its presence in a shipper's products. The interpretation of all subsequent corrosion data is complicated by the presence of a multiplicity of additives in the products of numerous shippers.

In the spring of 1954, the major user of the pipeline introduced a new proprietary corrosion inhibitor into some of its products. This shipper furnishes about 70 percent of the total volume of products transported. At this time, injection of Oronite R and T corrosion inhibitor into this shipper's products was discontinued. No significant change in corrosion rates was noted. The use of Oronite R and T corrosion inhibitor was discontinued entirely toward the end of 1954 on a trial basis. Subsequent corrosion measurements showed that satisfactory control was being accomplished by the presence of inhibitors in certain shippers' products. No further changes have been made in the pipeline corrosion inhibitor program since the fall of 1954.

Summary of Corrosion Measurements

A detailed summary is presented in Table 2 of corrosion measurements made during the last few years. Included in the table is an estimate of the percentage of the products containing corrosion inhibitors to which each test coupon was exposed. It is believed that all of these inhibitors are quite effective in reducing corrosion. Since there is no convenient method for determining the relative effectiveness of the individual corrosion inhibitors, all products containing such materials are treated collectively in

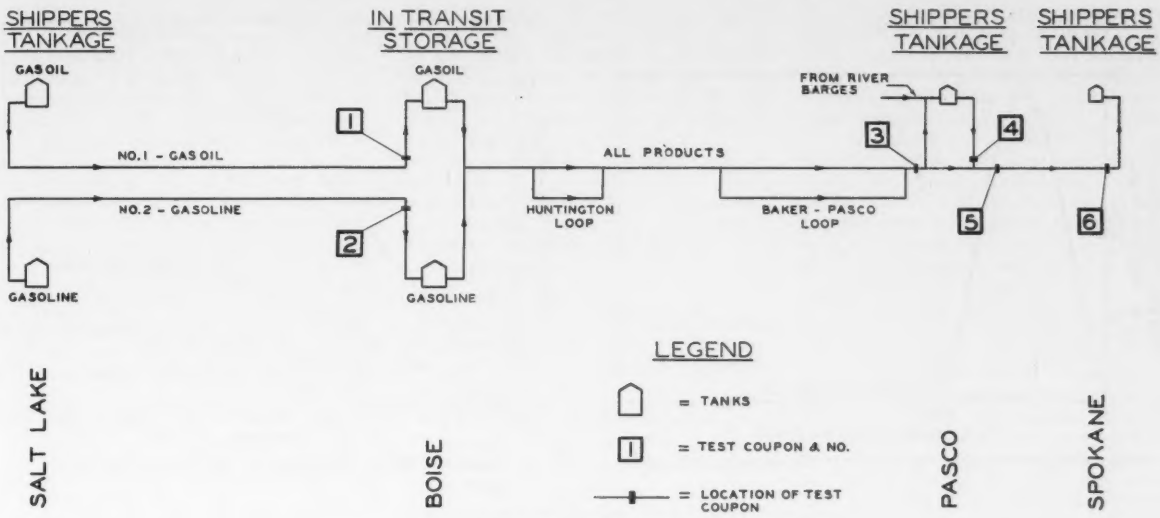

Figure 4—Flow diagram of products pipeline.

TABLE 1—Corrosion Rates in Early Operation of Pipeline

Date	Mode of Operation	Average Corrosion Rate, mil/year
6/50-9/51....	Alkaline sodium nitrite injected into motor gasolines.	0.15
9/51-4/54....	Oronite R and T corrosion inhibitor injected into motor gasolines.	0.04
4/54-11/54...	New proprietary additive present in the major shipper's gasolines. Oronite R and T injected into all other motor gasoline.	0.04
11/54 to date..	Use of Oronite R and T corrosion inhibitor discontinued.	See Table 2

Table 2. The estimate of the total percentage of the products which contain corrosion inhibitors is subject to some uncertainty because it is not always known which additives, if any, are utilized in the various products of different shippers. It is believed, however, that any errors due to this source are small. At least four different oil soluble corrosion inhibitors were separately contained in the products transported during this period.

Because of the overlapping exposures of the coupons, each succeeding measurement of the corrosion rate acts as a check on the corrosion rate determined from the preceding coupons. Therefore, an abnormally high or low rate must be substantiated by a succeeding measurement to be considered significant. Inspection of the data in Table 2 indicates nearly all results to be valid. The data establish that very low corrosion rates are occurring in the pipeline. A comparison of the results obtained from one location with those of another location is not of great significance because of the differences in the environments which produce the corrosion. Most of the test coupons showed corrosion rates of less than 0.04 mil per year. However, during a six-month period about the end of 1955, a corrosion rate of 0.1 mil per year was measured in the incoming line at Pasco. This coincided with the time when the lowest proportion of corrosion inhibited products passed through this location. This indicates that at this location more than 50 percent of the products must be inhibited to obtain maximum pipeline protection.

The smallest proportion of inhibited products going through any portion of the pipeline was generally that coming from the shippers' tanks at Pasco. Here, for prolonged periods, it is believed that less than 20 percent of the products contained corrosion inhibitors. However, the average corrosion rate of less than 0.03 mil per year measured in this stream indicates that the corrosive environment in this location was mild.

The statistical distribution of the data in Table 2 has been plotted in Figure 5 and shows a distribution about the value of 0.025 mil per year. From the data of this graph and the measured value of 1.2 mils per year corrosion obtained when no inhibitor was present in the pipeline, it is concluded that the present inhibitor program is approximately 98 percent effective. This degree of protection is obtained even though there are prolonged periods when no inhibited product is present at any given point. Visual inspection of the internal surface of piping opened up for alteration projects confirms the spectacular performance of the inhibitors. Such surfaces are clean and rust free.

Aircraft Fuels

A problem relating to the use of corrosion inhibitors arose when JP-4 jet fuel and aviation gasoline were first transported through the pipeline. Both of the products were for use in military aircraft and carried rigid water tolerance specifications. Without going into the details or merits of these specifications and tests, it may be stated briefly that "water tolerance" is a measure of the ability of the product to separate itself quickly and completely from water. These aircraft fuels, after being transported through the pipeline, no longer satisfied the water tolerance specification. An extensive and careful investigation eventually showed the difficulty to be caused by corrosion inhibitors desorbed from the surfaces of the pipe. Control was accomplished by ensuring that products free of corrosion inhibitor preceded the jet fuel and aviation gasoline batches.

The portions of the pipeline system put in service after September 1951 have been protected only through the use of oil soluble inhibitors. No important quantities of mill scale have been loosened from the interior of such portions of the pipe. During the first two or three months of operation of a newly constructed section of pipeline, scrapers were run as frequently as once per week in gasolines to remove the last traces of the water used for hydrostatic testing which may be left in sags and low places and to clean out the remainder of construction debris. Some moderate quantities of mill scale were brought in also. Thereafter, scrapers were run at three-month intervals only as an additional check on the internal condition of the pipe as compared with the corrosion rate shown by the test coupons. This is in contrast with previous experience in starting up a new line using a water soluble corrosion inhibitor. The introduction of a water soluble corrosion inhibitor into a new line necessitated excessively frequent scraper running and the disposal of huge quantities of rust and mill scale.

The present concept of an acceptable maximum corrosion rate in the Salt Lake pipeline is 0.05 mil per year. Experience has shown that control to this limit is a practical and economical accomplishment. This corrosion rate is equivalent to a reduction in pipe wall thickness of 0.001-inch in 20 years, a loss of metal which is obviously of no practical concern. However, when corrosion rates exceed about 0.05 mil per year, some rust

Figure 5—Statistical distribution of corrosion measurements (1955-1957).

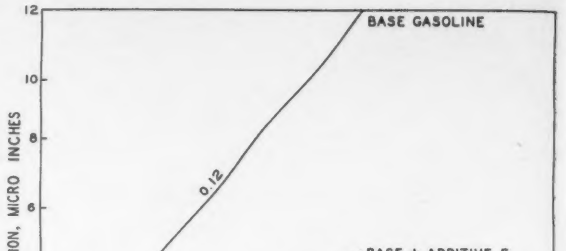

Figure 6—Inhibition of corrosion of a sand blasted steel surface.

TABLE 2—Corrosion Rates At Six Locations

Period of Specimen Exposure	No. 1 (Boise Line 1)		No. 2 (Boise Line 2)		No. 3 (Pasco Incoming Line)		No. 4 (Pasco Line from Tankage)		No. 5 (Pasco Outgoing Line)		No. 6 (Spokane Terminal)	
	Corrosion Rate (mpy)	Est. % of Inhibited Products	Corrosion Rate (mpy)	Est. % of Inhibited Products	Corrosion Rate (mpy)	Est. % of Inhibited Products	Corrosion Rate (mpy)	Est. % of Inhibited Products	Corrosion Rate (mpy)	Est. % of Inhibited Products	Corrosion Rate (mpy)	Est. % of Inhibited Products
Mar. 57-Sep. 57	0.02	63	0.03	74	0.04	83	0.01	20	0.07	68	0.08	68
Dec. 56-Jun. 57	0.01	57	0.02	67	0.04	77	0.02	27	0.04	73	0.02	62
Sep. 56-Mar. 57	0.03	47	0.02	58	0.04	66	0.03	21	0.03	55	0.04	53
Jun. 56-Dec. 56	0.01	40	0.01	64	0.04	72	0.03	17	0.03	59	0.05	58
Mar. 56-Sep. 56	0.01	68	0.01	67	0.03	74	0.02	16	0.01	51	0.03	56
Dec. 55-Jun. 56	0.02	37	0.02	56	0.04	57	0.02	7	0.03	51	0.03	50
Sep. 55-Mar. 56	0.02	40	*	53	0.11	51	0.04	16	0.03	46	0.04	47
Jun. 55-Dec. 55	0.02	38	0.04	46	0.09	42	0.03	34	0.02	54	0.02	53
Mar. 55-Sep. 55	0.01	39	0.01	61	0.03	63	0.02	21	0.03	53	0.03	53
Dec. 54-Jun. 55	0.03	30	0.03	66	0.02	64	0.02	17	0.02	49	0.02	49

* Coupon damaged by scraper.

may be observed on the test coupons. A scraper may traverse a section of line 100 miles or more in length and the resulting accumulation of rust may serve as a source of contamination of products. The rust may be extremely slow in settling, necessitating extra tankage for storage of products. Also, any appreciable rust scale on the internal surface of the pipe increases friction, resulting in reduction of throughput capacity and wastage of power.

Mechanism of Corrosion Inhibition

The conditions which cause corrosion in the pipeline vary in a highly complex fashion throughout the line. Many products are transported. Some of these products are free of corrosion inhibitors, while other products may separately contain any one of several inhibitors. Velocities and degrees of agitation vary throughout the pipeline. Before discussing the corrosion occurring in a pipeline, it is desirable to describe briefly the mechanism by which corrosion inhibitors function under somewhat simplified conditions.

An oil soluble corrosion inhibitor is effective because it produces an adsorbed film on the surface of the metal. This film decreases contact of corrosive agents with the metal surface. The geometry and size of the adsorbed molecules and the manner in which the electric charge is distributed govern the effectiveness of inhibitors. They diminish the rate at which all molecular species and ions can contact the metal surface.[4] Thus, they act as impermeable surface films in a manner analogous to an ordinary coat of paint.

A concise review and discussion of the role of adsorption in inhibitor action was presented by Hackerman and Schmidt,[5] whose conclusions are liberally quoted in the next few paragraphs. The adherence of a surface active corrosion inhibitor to a metal may be due to purely physical forces, as in physical adsorption, or the surfactant may be held more strongly by chemical forces, as in chemisorption. The difference between these two processes may be considered one merely of degree. In extreme cases, however, these two types of adsorption phenomena may be distinguished by differences in ease of reversibility, temperature coefficients, and heats and rates of adsorption. Both types of adsorption occur by association of the polar portion of the inhibitor molecule with the surface of the metal. When the inhibitor is present in sufficient concentration, the surface becomes covered with a close-packed impervious film of inhibitor molecules. With the highly effective inhibitors that are available today, inhibitor concentrations of only a few parts per million are required to control the corrosion.

Not only is the extent of coverage important in inhibiting corrosion, but also the type of coverage determines the effectiveness. Physically adsorbed substances retard corrosion primarily by decreasing the ability of the corrosive molecules to reach the metal surface. A characteristic of physical adsorption is the nearly instantaneous formation of the adsorbed film. In chemisorption, the adsorbed material satisfies secondary valence forces at the surface. Consequently, chemisorbed molecules have far less tendency to redissolve than molecules held only by physical forces. In addition, the chemisorbed coating reduces the tendency of the atoms of the metal surface to leave the crystal lattice.

The tendency of a surface active agent to be chemisorbed does not necessarily increase with decreasing temperature, as it does in physisorption. The temperature coefficients of chemisorption are complex and not readily predictable.[6] The effectiveness of chemisorbed corrosion inhibitors may either increase or decrease as the temperature is increased. This is one reason why the exact behavior of corrosion inhibitors in practical systems, such as pipelines, is very difficult to predict.

Inhibitors exhibit their optimum effect when the entire surface of the metal is covered with molecules held by chemical forces. "The metal then is less likely to dissolve because (a) the activity of each atom on the surface is decreased, (b) the corrosive agent is retarded in its approach to the surface, and (c) the inhibitor would not be removed easily by re-solution in the surrounding liquid."[5] However, in the practical case, the metal is never covered entirely by chemisorbed inhibitor molecules. Probably chemisorption occurs on the more active portions of the metal, while most of the remainder of the surface is covered by molecules held by the weaker physical forces. The active portions of the metal are areas which have the greatest tendency to be attacked by corrosive agents. Thus, the portions of the surface which are most readily corroded are those which are most easily inhibited by chemisorption.

Since the adsorption processes are reversible, Hackerman states that an inhibitor must be renewed continuously for it to be effective, particularly if there is continuous flow of the liquid past the metal.[5] Data on the application of corrosion inhibitors in pipelines show that the continuous renewal is not necessary, although periodic renewal obviously is.

Theories of adsorption do not require that total coverage with inhibitor molecules be obtained on solid surfaces. In the case of relatively close-packed molecules, however, lateral forces between the hydrocarbon chains can function to provide additional protection. In systems containing hydrocarbons, nonpolar molecules can be oriented in the adsorbed film, thereby providing more complete coverage. The forces operating between neighboring physically adsorbed and chemisorbed molecules reduce the tendency of the former to redissolve. The magnitude of the forces in such lateral action is the same as the forces causing physical adsorption. In this way, coverage is provided for the portions of the surface on which adsorption of the inhibitor does not occur. It thus becomes more difficult to remove the less firmly held inhibitor from the surface.[5]

In the practical case of the pipeline, products having grossly different chemical composition are transported. Polar impurities are present, and the hydrocarbon constituents vary over a wide range. Thus, the nonpolar molecules which become oriented on the surface change as the fuel composition is varied. This is one of the factors which accounts for the difference in effectiveness of various corrosion inhibitors in different samples of a given petroleum product. Additionally, it is postulated that the polar impurities in the product compete with the corrosion inhibitor for reactive sites on the surface. Such adsorbed polar impurities probably are another cause of the somewhat erratic response of petroleum products to corrosion inhibitors.

Laboratory Evaluation of Adsorption and Desorption

In interpreting the data from the pipeline, it is necessary to employ conclusions reached in laboratory experiments. Laboratory tests provide a convenient means for studying the mechanism of corrosion inhibition but should not be regarded as a method for the quantitative comparison of the effectiveness of pipeline corrosion inhibitors. The relative activities of inhibitors are too dependent on test conditions to justify such quantitative laboratory comparisons. The following variables are important in evaluating corrosion inhibitors:

1. Temperature
2. Condition of surface
3. Amount of corrosive agent present, generally water
4. Ions present in the aqueous phase
5. Degree of agitation
6. Particle size of any dispersed phase
7. Amount and type of polar compounds present as impurities in the hydrocarbon phase
8. Composition of the hydrocarbon
9. Properties of the corrosion inhibitor
10. Concentration of inhibitor

The adsorbed film of inhibitor may be considered to be in equilibrium with the inhibitor dissolved in the hydrocarbon. All surfaces compete to adsorb the surface active agent. Hydrocarbon-water interfaces act as surfaces, competing with the surface of the metal for corrosion inhibitor molecules.

When a fuel containing a corrosion inhibitor contacts the surface of the pipeline, an equilibrium film of inhibitor is adsorbed on the surface of the pipe. In the case of a petroleum product free of corrosion inhibitor following an inhibited stock through the pipeline, the equilibrium concept leads to the prediction that the inhibitor should desorb into the uncompounded product causing the surface protection gradually to disappear. The rate of removal of the corrosion inhibitor will be affected by the magnitude of the forces holding the adsorbed layer.

Behavior of Adsorbed Films in Pipeline

The following laboratory experiments were conducted to demonstrate the behavior of adsorbed films in the pipeline. In these experiments, a "Corrosometer" manufactured by the Crest Instrument Company (Santa Fe Springs, California) was employed to measure quantitatively the corrosion of a mild steel ribbon. The technique used in measuring these rates was similar to that described by Stormont[7] and by Rohrback.[8]

In the first set of experiments, the results of which are shown in Figure 6, sand-blasted steel surfaces were subjected to corrosion in a mixture of synthetic sea water and a commercial premium grade gasoline. At room temperature, 50 ml of the synthetic sea water and 250 ml of the gasoline were agitated in a Berzelius beaker containing the test steel specimen. The commercial base gasoline employed contained no corrosion inhibitor. Four test additives were utilized in this and succeeding experiments. All of these additives have been used commercially as corrosion inhibitors and proved to be highly effective. The principal active ingredient of each additive may be categorized as follows: Additive "D" is a lead sulfonate; Additive "E" is an amine derivative of a sulfonate; Additive "F" is an amine-type corrosion inhibitor; and, Additive "G" is primarily a carboxylic acid-type inhibitor. These inhibitors were not all used at the same concentration. It should be emphasized that the experiments were not designed to compare the efficiency of the additives, but rather to demonstrate the properties and mode of action of corrosion inhibitors.

The slopes of the curves of Figure 6 correspond to the rates of corrosion of the metal surface in microinches per minute. It is seen that the base gasoline plus sea water produces a rate of attack of 0.12 microinch per minute. Addition of the corrosion inhibitors reduced the rate of attack of the sand-blasted steel surface to between 0.01 and 0.045 microinch per minute. This reduction in corrosion is far less than the reduction realized in the pipeline, owing to the extremely severe test conditions employed.

In a second set of experiments, results of which are shown in Figure 7, inhibition of corrosion of a rusted steel surface is illustrated. In these experiments, the sand-blasted steel surface was first permitted to rust for 40 minutes. In the plots, the time scale begins at 20 minutes because a few minutes are required for the corrosion to attain a constant rate. After 40 minutes of contact with uncompounded gasoline and sea water, the uncompounded fuel was replaced with a beaker of sea water and gasoline containing additive.

The data from Figures 6 and 7 are summarized as relative rates in Table 3. All of the inhibitors are far less effective on the rusty surface than on the fresh sand-blasted surface. Additive "F" appears to cause an increase in the rate of corrosion, but this increase is within the experimental error. These data serve to illustrate the importance of the condition of the surface of the metal in determining the effectiveness of corrosion inhibitors. As mentioned above, the conditions of this test are extremely severe. It is known that all of the test additives are highly effective corrosion inhibitors for a pipeline.

The equilibrium concept of the adsorbed film leads to the prediction that when the inhibitor no longer contacts the metal, the effectiveness of the corrosion inhibitor should gradually decrease. This effect is illustrated by the data plotted in Figure 8. Here, the gaso-

TABLE 3—Effectiveness of Corrosion Inhibitors in Commercial Gasoline

Corrosion Inhibitor Present	Relative Rates of Corrosion	
	Sand-blasted Surface	Rusted Surface
None	100	100
Additive "D"	21	54
Additive "E"	21	35
Additive "F"	37	108
Additive "G"	8	33

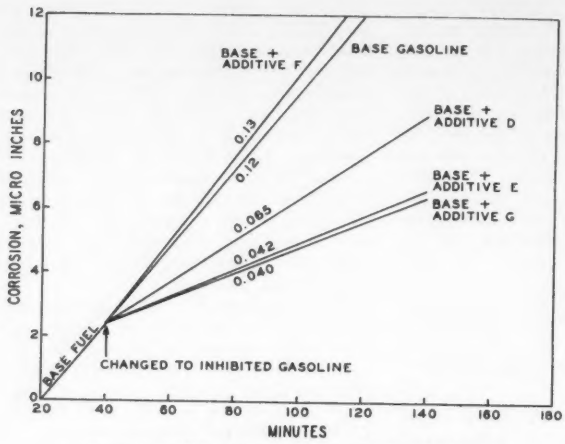

Figure 7—Inhibition of corrosion of rusted steel surface.

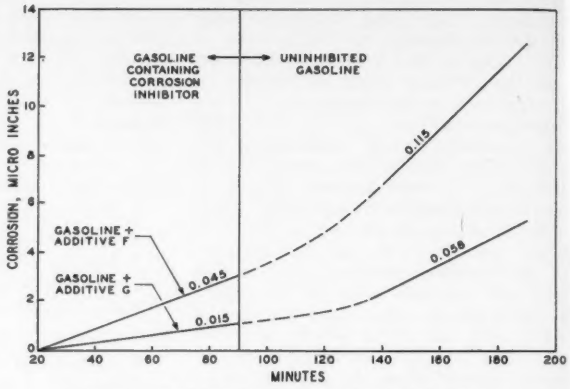

Figure 8—Effect of discontinuous use of corrosion inhibitor.

lines containing corrosion inhibitors first contacted the sand-blasted steel strips for 90 minutes. The corrosion inhibited gasolines were then removed and replaced with uninhibited fuel. The corrosion rates then gradually increased. Within about one hour, following replacement of the gasoline containing Additive "F" with the noncorrosion inhibited gasoline, the rate returned to that which existed when no inhibitor had ever contacted the metal. Following replacement of the fuel containing Additive "G" with the uninhibited gasoline, a new corrosion rate of 0.06 microinch per minute was established, within about an hour. This new rate remained constant for several hours. It is presumed that if this experiment had been conducted long enough, the rate of corrosion would eventually have approached the rate of the base gasoline. The new rate of about 0.06 microinch per minute indicates the presence of a very stable chemisorbed film of corrosion inhibitor. It should be emphasized that the conditions of these experiments are extremely severe. In the milder environment in the pipeline, it is anticipated that a much longer time would be required for significant quantities of the corrosion inhibitor to be desorbed. The fact that a great reduction in corrosion of the pipeline occurred, even though products free of corrosion inhibitor were transported for periods of days, substantiates this prediction.

There remains to be considered the question of what happens when an inhibited product is followed by a product containing another inhibitor. As was shown above, when an uninhibited product is transported past a surface coated with an adsorbed inhibitor, much of the corrosion inhibitor gradually desorbs into the solvent and presumably the tightly chemisorbed material eventually will be removed. Desorption should be aided by the presence of polar materials which are impurities in the product and compete with the corrosion inhibitor for the surface. When a second corrosion inhibitor is present in the product, it is reasonable to presume that this latter inhibitor would exert additional force in desorbing the first inhibitor. Thus the second inhibitor may be expected to replace the physisorbed portion of the first inhibitor rapidly and the more tightly held chemisorbed portion at a slower rate. Thus, in products transported in the pipeline, there is some interchange of corrosion inhibitors and other surface active agents from one stock into another.

Displacement of Inhibitor

Experiments were conducted using the "Corrosometer" to demonstrate the effect of one corrosion inhibitor displacing another. It was qualitatively observed that displacement of inhibitors did occur. Frequently this displacement was not complete, at least during several hours of observations. In such cases, it is postulated that the resultant adsorbed film of corrosion inhibitor consisted of a mixture of molecules. Such mixed films differed considerably in effectiveness from those obtained with either inhibitor alone. Use of more than one inhibitor in a pipeline, therefore, does not necessarily give better protection against corrosion than would be obtained with a single inhibitor, and may result in poorer protection. However, it is believed that the currently available oil soluble inhibitors are so effective that any combination used in a petroleum products pipeline probably will give adequate protection.

The desorption of a corrosion inhibitor from the metal surface into a product and the displacement of one inhibitor by another explains the difficulty mentioned previously with the water tolerance properties of aircraft fuels transported through the pipeline. Apparently, corrosion inhibitors were being introduced into these fuels by desorption, causing the fuels to fail the water tolerance test. Complex mixtures of surface active agents frequently act as emulsifying agents. The problem was solved by the practice of preceding the aircraft fuels with a gasoline free of corrosion inhibitor. This resulted in desorption of any loosely held corrosion inhibitor into this gasoline, protecting the aircraft fuels.

Conclusion

A water soluble corrosion inhibitor, sodium nitrite, has been compared with oil soluble corrosion inhibitors in the same pipeline system. The former material was about 90 percent effective in controlling corrosion, but its use required frequent running of scrapers and the disposal of large quantities of rust and mill scale. The rust and scale introduced into the petroleum products caused problems due to the slow settling of the solids and water from the products. Also, the use of the water soluble inhibitor was largely responsible for water cloud problems during cold weather.

Oil soluble corrosion inhibitors were shown to be very effective in the pipeline, reducing the corrosion rate to about 0.025 mil per year and minimizing the settling problems encountered with the nitrite. This very low rate of corrosion was obtained in spite of the fact that prolonged periods occurred when no inhibited product contacted sections of the pipeline.

Oil soluble corrosion inhibitors are known to function because they are adsorbed on surfaces. The inhibitors adhere to metals partly due to physical forces and partly due to the stronger chemical forces which occur in chemisorption. The physically adsorbed inhibitors are relatively rapidly desorbed on contact with a fuel containing no corrosion inhibitor, but the chemisorbed molecules are removed only very slowly resulting in prolonged protection of the metal from corrosion.

The use of more than one oil soluble corrosion inhibitor in a pipeline causes mixtures of inhibitor molecules to be adsorbed on the surface of the pipe. One inhibitor tends to displace another. This results in some interchange of the corrosion inhibitors among the products transported. The use of several corrosion inhibitors in a pipeline may give better or poorer protection than would be obtained with a single inhibitor. However, the currently available oil soluble inhibitors are so effective that most combinations used in a products pipeline will give adequate protection.

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DISCUSSION

Questions by Ivy M. Parker, Plantation Pipe Line Co., Atlanta, Georgia:

What was the time interval between starting operation of pipeline and starting of inhibitor in June, 1950?

We feel that the idea that all mill scale must be removed from a pipeline is wrong and that if inhibitor concentration is developed immediately when the pipeline is put into operation, there will be no need to handle large volumes of scale nor to run an excessive number of scrapers.

Late in 1951, we placed two pipelines in operation with sodium nitrite as inhibitor. In one, an 18-inch line, we did not get the inhibitor concentration up to required amount until two years had elapsed. Large quantities of sediment were removed from this line (see Figure 9). In the other line (14-inch), the inhibitor concentration was brought up quickly and a relatively small amount of sediment was removed (see Figure 10).

It would be interesting to know the average nitrite content of the effluent water at the point where the corrosion coupons showed 0.15 mpy, as shown in Table 1.

Reply by M. R. Barusch

The first portion of the pipeline was placed in operation in January 1950. All the line to Pasco was in service by the end of September 1950. Sodium nitrite solution was introduced into the line in June, so that some portions of the line were contacted with nitrite at the time of their initial service. We agree that it is desirable to introduce a corrosion inhibitor into a pipeline during its initial operation.

Data on average nitrite contents of the water at the locations where the corrosion coupons were mounted are not available. Efforts were made to maintain a minimum content of 1 percent sodium nitrite and a pH of 8.0 in the water effluents from the pipeline. Generally, the sodium nitrite content in the effluent was 1-2 percent and the pH was 8.0 to 9.5.

Comments by Albert W. Jasek, Humble Pipe Line Company, Houston, Texas:

It is interesting to note that the authors report an experience which substantiates and confirms the experience my company has observed with sodium nitrite, a water soluble inhibitor, and then with an oil soluble inhibitor. My company's experience is in agreement with the authors report in the following respects.

(1) Oil soluble inhibitors provide a greater degree of rust protection than that observed with the use of water soluble sodium nitrite.

(2) The frequency of scraping the line was reduced considerably after using the oil soluble inhibitor.

(3) Uninhibited products tend to wash off the film deposited by the inhibited products; however, it is still possible to move batches of uninhibited aviation gasoline through the pipe line system and maintain control of internal corrosion. The author's investigations indicate why this is possible.

The installing of steel coupons for evaluating corrosion control appears to have some merit and in connection with this method of evaluating corrosion, the NACE Committee T2-E is sponsoring a coupon testing program to secure data which would indicate the relative effectiveness of the different types of inhibitors used by various product pipe lines.

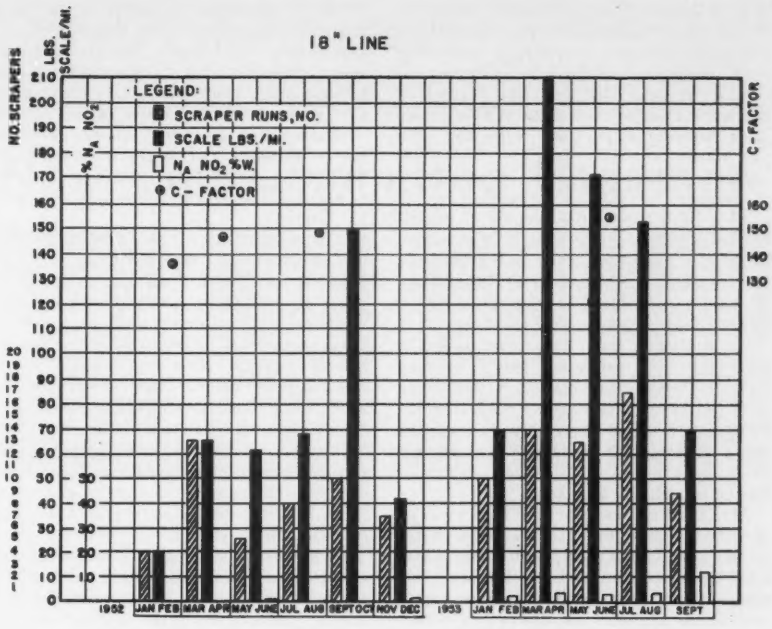

18" LINE
LEGEND:
SCRAPER RUNS, NO.
SCALE LBS./MI.
$N_A NO_2$ %W.
C- FACTOR
NO. SCRAPERS
LBS. SCALE/MI.
% $N_A NO_2$
C-FACTOR
1952 JAN FEB MAR APR MAY JUNE JUL AUG SEPT OCT NOV DEC 1953 JAN FEB MAR APR MAY JUNE JUL AUG SEPT

Figure 9—Resume of scale collected in hay tanks, scraper runs, inhibitor and "C" factor for 18-inch line.

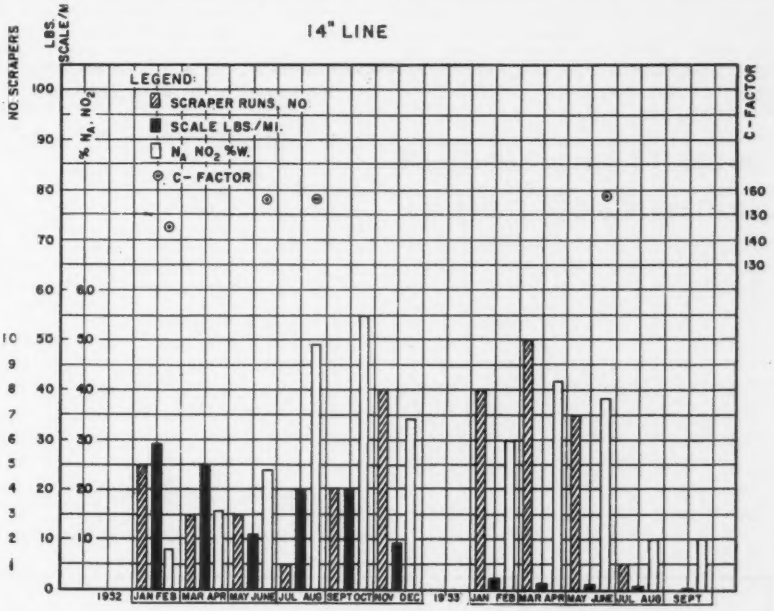

14" LINE
LEGEND:
SCRAPER RUNS, NO
SCALE LBS./MI.
$N_A NO_2$ %W.
C- FACTOR
NO SCRAPERS
LBS. SCALE/MI
% $N_A NO_2$
C-FACTOR
1952 JAN FEB MAR APR MAY JUNE JUL AUG SEPT OCT NOV DEC 1953 JAN FEB MAR APR MAY JUNE JUL AUG SEPT

Figure 10—Resume of scale collected in hay tanks, scraper runs, inhibitor and "C" factor for 14-inch line.

It is believed that the best indication of the effectiveness of a corrosion inhibitor is the amount of sediment and other solid material removed by filtering. It would be interesting to know whether the authors have any data which correlates filtering experience with the coupon data.

Another factor which is pertinent to the control of internal corrosion by oil soluble inhibitors is the pounds of inhibitor required per 1000 barrels of product. It would certainly be of value to have information which correlates the amount of inhibitor required to reduce corrosion to the degree shown in Figure 7.

The authors report some difficulty with the water tolerance test. According to tests made by our company, the amine type inhibitors generally affect water tolerance test more adversely than other types of inhibitors which we investigated.

Reply by M. R. Barusch:

Mr. Jasek states "the best indication of the effectiveness of a corrosion inhibitor is the amount of sediment and other solid material removed by filtering." Indeed, the quantities of sediment so removed by strainers or filters are highly significant. However, in the Salt Lake products pipeline, a portion of such material originates from shippers' tanks and lines, with some foreign material being introduced by construction and maintenance work on the system. It is our opinion that in this pipeline, test coupons offer a more reliable method for evaluating corrosion; consequently, no efforts have been made to collect quantitative data on the solids removed by filtering.

The concentrations of corrosion inhibitor present when obtaining the data of Figures 6, 7 and 8 varied from 20-50 ppm. It should be emphasized, however, that these experiments were designed to demonstrate the behavior of corrosion inhibitors rather than to measure their effectiveness. Slight changes in any of several test variables would greatly alter the relative effectiveness of the inhibitors tested. We believe that the only reliable way of determining the minimum amount of inhibitor required to control corrosion in a pipeline is to experiment in the pipeline itself.

Most of the corrosion that occurs in the subject pipeline probably takes place when stocks free of corrosion inhibitor are in contact with the surface of the pipe. During this time, the corrosion inhibitor which is held by physisorption desorbs relatively rapidly, and the chemisorbed inhibitor is removed slowly. When Oronite R and T corrosion inhibitor was added to the gasolines transported through the Salt Lake product pipeline, it was usually added at a concentration of 50 ppm. No attempts were made to determine the minimum amount of corrosion inhibitor which could have been used. However, on a trial basis the concentration of the R and T inhibitor was doubled, and no significant effect on corrosion rate was observed. This observation is consistent with the theory of how corrosion inhibitors function. An increased concentration of corrosion inhibitor would tend to increase the amount of corrosion inhibitor adsorbed on the surface of the metal. Higher concentrations of corrosion inhibitors would tend to displace other polar materials or solvent molecules adsorbed on the surface. The resultant closer-packed film of corrosion inhibitor would contain additional inhibitor molecules held by forces of physisorption. The additional inhibitor molecules adsorbed would be relatively rapidly removed by uninhibited stocks and therefore would provide little additional protection against corrosion.

Any discussions of this article not published above will appear in the June, 1959 issue

DISCUSSIONS ON TECHNICAL ARTICLES ARE SOLICITED

Discussions on technical articles published in Corrosion will be accepted for review without invitation. Discussions must be constructive, accompanied by full substantiation of fact in the form of tables, graphs or other representative data and be submitted in three typewritten copies. In cases where illustrations are submitted, at least one copy of figures should be of a quality suitable for reproduction.

Authors will be sent on request a copy of the NACE Outline for the Preparation and Presentation of Papers.

Discussions will be reviewed by the editor of Corrosion and will be sent to the author of the paper discussed for his replies, if any. Publication will be in the Technical Section with full credit to the authors together with replies. Discussions to papers presented at meetings of the association may be submitted in writing at the time of presentation or later by mail to the editorial offices of Corrosion, 1061 M & M Bldg., Houston 2, Texas.

TECHNICAL COMMITTEE ACTIVITIES

Metallurgy Committee Prepares Agenda For Chicago Meeting

Proposed agenda for the Chicago meeting of T-1F Unit Committee on Metallurgy has been mailed to committee members for comments. The meeting will be Thursday afternoon, March 19, in Sherman Hotel's Gold Room during the 15th Annual Conference in Chicago.

Items on the agenda are as follows:

1. Alternate string sucker rod tests.
2. Alternate string coupling tests.
3. API Committee on corrosion fatigue testing.
4. Materials for high temperature production.
5. Monel sucker rod tests.
6. Research projects.
7. T-1F-1 Chairman: F. A. Prange.
8. T-1F-2 Chairman: J. P. Fraser.
9. T-1F organization.

Ten committee members and 36 guests were present at the New Orleans meeting held in October.

Topics discussed at the meeting included alternate string sucker rod tests and case hardened sucker rod couplings. Also, reports were given by each task group.

Fourth Meeting Held By T-8 Committee

Fourth meeting of T-8 Technical Committee on Refining Industry Corrosion held last October in New Orleans was attended by 90 people. Nine new members were added since the March, 1958, meeting.

Topics discussed at the two-day meeting included corrosion in reforming and desulfurization units, corrosion of exchangers and condensers, naphthenic acid corrosion, chemical cleaning, corrosion during downtime and effect on corrosion of using chlorinated solvents.

Other topics were corrosion in amine treating units scrubbing gases from catalytic cracking and metallurgical phenomena of interest to refinery corrosion engineers.

New topics discussed were corrosometer application, corrosion in isomerization units and experiences with Cronox C inhibitor.

Prime

VICE CHAIRMAN for Group Committee T-4 on Utilities is Joseph B. Prime, Jr., Florida Power and Light Co., Miami. He has been a member of NACE since 1950. His work involves design and maintenance of cathodic protection equipment for plant and transmission distribution cables.

New Unit Committee Chairmen

Burns

Broyles

NEW OFFICERS for T-2H Committee on Asphalt Type Pipe Coatings have been elected. Chairman is Frank B. Burns, General Asphalts, Inc., Wynnewood, Okla., and vice chairman is Wayne B. Broyles, Wayne Broyles Engineering Corp., Houston. Mr. Burns, a NACE member since 1954, has a BA from Vanderbilt University. He is secretary-treasurer for the Central Oklahoma Section. Mr. Broyles is a corporate member and has been active in NACE since 1948.

Brannon

Nelson

NEW CHAIRMEN for T-2J Committee on Wrappers for Underground Pipe Line Coatings are Chairman Russell A. Brannon of Humble Pipe Line Co., Houston, and Vice Chairman Loyd B. Nelson of Shell Pipe Line Corp., Houston. Mr. Brannon, one of the founders of NACE, was elected as the first president and was treasurer from 1950 to 1957. He is supervising corrosion engineer at Humble and has been with the company since 1928. Mr. Nelson, active in NACE since 1944, is senior corrosion engineer at Shell. A graduate of Colorado University, he has been with Shell since 1933.

Coordinating Committee Votes for Name Change

Southern West Virginia Corrosion Coordinating Committee is the new name for the Charleston Coordinating Committee affiliated with T-7A, Northeast Region Corrosion Coordinating Committee. Members voted for the change at a meeting in Charleston last November.

New officers for the committee were elected also: Chairman K. R. Gosnell, Vice Chairman P. F. Sweeney and Secretary J. A. Parker.

Representatives from each participating company gave brief descriptions of rectifier installations now operating or proposed.

T-3F Committee Discusses Current Corrosion Problems

A discussion of current corrosion problems was held as part of the Cincinnati meeting Unit Committee T-3F, Corrosion by High Purity Water last October.

The discussion included stress corrosion cracking in relatively high purity water, corrosion of zirconium, oxygen scavengers and the metallurgy of some materials used in high purity water.

Research problems worthy of committee sponsorship were discussed. A style sheet has been mailed to members as a guide for submitting possible corrosion research problems.

•

Over 70 technical committees will meet during the 15th Annual NACE Conference in Chicago.

•

Over 226,000 copies of the NACE technical committee reports have been distributed.

Burgess and Dietze To Be T-4F Chairmen

New chairmen for Unit Committee T-4F on Materials Selection for Corrosion Mitigation in the Utility Industry have been elected. They are Chairman W. T. Burgess and Vice Chairman I. C. Dietze.

Mr. Burgess is corrosion engineer in the department of design and operating engineering of the Metropolitan Utilities District, Omaha, Nebraska. He attended University of Nebraska. His corrosion work has been in surveys, tests, drains and anode installations. He has been an active member of NACE since 1949.

A graduate of the University of California, Mr. Dietze has been a member of NACE since 1945. He is an electrical engineer with the department of water and power of the City of Los Angeles. He has served as chairman of Technical Group Committee T-4 on Corrosion in the Utilities Industry and has been chairman and program chairman of the Western Regional Division.

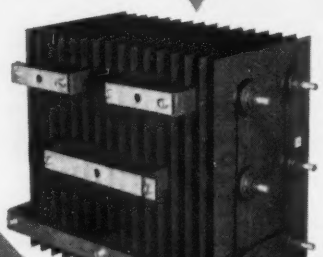
T-8A Committee Meets

Task Group T-8A on Chemical Cleaning held a meeting in New Orleans as part of the South Central Region Conference last October.

Case histories of 11 chemical cleaning operations were presented and discussed to determine what control and supervision are necessary for satisfactory results and minimum damage to equipment.

Report was given on a survey of what refinery areas make most use of chemical cleaning techniques.

at the heart of their CATHODIC PROTECTION EQUIPMENT...leading manufacturers use



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*HIGH CURRENT DENSITY

for increased efficiency, far longer life, and lower operating costs

Throughout the U. S. for nearly two years now, cathodic protection equipment using RRco. high current density stacks have been giving continuous and trouble-free service – including applications with the most severe surge conditions. It's a repeat of the amazing record these cells established long ago in European applications.

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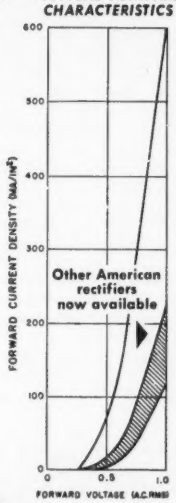
When you need new cathodic protection equipment, make sure it's *really* modern. See that it contains Radio Receptor HCD PETTI-SEL or TRI-AMP Rectifiers.

TYPICAL FORWARD CHARACTERISTICS



FREE INFORMATION . . . Leading pipeline companies are now using Petti-Sel and Tri-Amp stacks as standard equipment for cathodic protection. For a list of users, as well as further information on these important products, write today to Section C-3.

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NACE NEWS

15th Annual Conference Plans Are Complete for March 16-20

Plans are completed for the 15th Annual NACE Conference to be held March 16-20 in Chicago's Hotel Sherman.

Ninety technical papers representing the work of about 130 authors on a wide diversity of corrosion control problems will be presented in 16 symposia during the conference.

Seventy-two technical committee meetings are scheduled also.

Advance program for the conference was published on pages 75-102 in January CORROSION with additions and corrections on pages 67 and 72 of the February issue.

The Inter Society Corrosion Committee will meet in the Life Room (Room 108) of Hotel Sherman from 2-5 pm, Thursday, March 19.

The Regional Management Committee will meet 2-5 pm, Tuesday, March 17, in the Gold Room (Room 114).

The Policy and Planning Committee meets Monday, March 16, from 2-5 pm in the Life Room (Room 108).

1959 Corrosion Show To be March 17-19

Diversified displays of corrosion control materials, equipment and processes will be featured at the 1959 Corrosion Show to be held March 17-19 in conjunction with 15th Annual NACE Conference in Chicago.

Over 90 companies will have displays in the 140 booths in the Hotel Sherman.

Exhibits will be staffed by experienced corrosion engineers who will be qualified to answer questions on corrosion control.

The show will be open from 10 am to 5 pm on each of the three days so that conference registrants will have an opportunity to visit the displays.

Three companies (Tret-o-lite Co., division of Petrolite Corp., Hercules Powder Co., and American Hot Dip Galvanizers) will have displays in addition to those companies listed on page 82, October CORROSION, page 95 of the December issue, page 82 of the January issue and page 72 of the February issue.

Error on H. T. Francis

H. T. Francis is electrochemistry research supervisor at the Armour Research Foundation, Chicago.

His company affiliation was published incorrectly on Page 100 of the January issue of CORROSION. He is one of the authors of a paper to be presented in the General Corrosion Symposium at the Chicago Conference this month.

Conference Registration At Chicago

Members who have not pre-registered will need to show their membership cards if they register at the membership rate for the 15th Annual NACE Conference at Hotel Sherman in Chicago. A schedule of the conference registration hours was published on page 83 of the January issue of CORROSION.

Punch Card Demonstration To Be Given at NACE Booth

A demonstration unit of the Corrosion Abstract Punch Card service will be located at the booth of National Association of Corrosion Engineers at the 1959 Corrosion Show. Miss Nicole Treves, who is in charge of issuing the punch cards at Central Office, will be present to explain the system and how it is being and can be used.

A principal aim this year will be to demonstrate the efficiency of sorting by use of the sorting machine, which permits using multiple needles in the selection device and makes use of the NACE random number system of topical indexing practical. This feature will be especially interesting to present users of the punch card service who do not already have the machine selector.

Miss Treves also will show the relationship between abstracts issued in the punch card service and those included in the bibliographic surveys of CORROSION.

NACE Publications to Be Displayed at Chicago Booth

The NACE booth at the 1959 Corrosion Show in Hotel Sherman, Chicago, during the 15th Annual Conference in March will have available inspection copies of all NACE publications. Orders for these publications will be taken by Central Office personnel at the booth.

Orders also will be accepted at the booth for advance printings of available conference papers.

NACE Central office staff members will be on hand to answer questions about the association, membership, technical committees and other matters.

Southeast Region News

Regional Officers as published on page 102 of December CORROSION omitted James L. English, Oak Ridge National Laboratory, as assistant secretary-treasurer. John B. West, Aluminum Company of America, Atlanta, is the director—not assistant secretary-treasurer as published.

Watson

Moorman

Spalding

INCOMING DIRECTORS

Representing Regions for three-year terms of office are T. R. B. Watson for the Canadian Region, E. F. Moorman for the North Central Region and J. C. Spalding for the South Central Region. Mr. Moorman is a research engineer in the Engineering Research Department of International Harvester Co., Chicago. He has a BA and MA from Indiana University. Mr. Watson, founder of Corrosion Service Limited, Toronto 5, Ontario, graduated in metallurgical engineering from the University of Toronto and has held several regional and sectional offices in NACE. Mr. Spalding of Sun Oil Co., Dallas, is a former faculty member of Southern Methodist University and has held several offices in the South central Region.

Russian Society Sends NACE New Year's Greetings

A letter extending good wishes to NACE from B. Shramchenko, deputy president of the All Union Council of National Technical Societies of the Soviet Union, was received recently by H. H. Uhlig, NACE member and professor of metallurgy at Massachusetts Institute of Technology.

The letter was translated as follows:

December 22, 1958
Moscow

Dear Professor Uhlig:

The All Union Council of National Technical Societies of the Soviet Union extends greetings to you and members of the Association for the coming New Year, 1959. We wish you and all members of the Association good health and further successes in the development of science and technology to the benefit of all mankind.

Sincerely yours,
B. Shramchenko
Deputy President

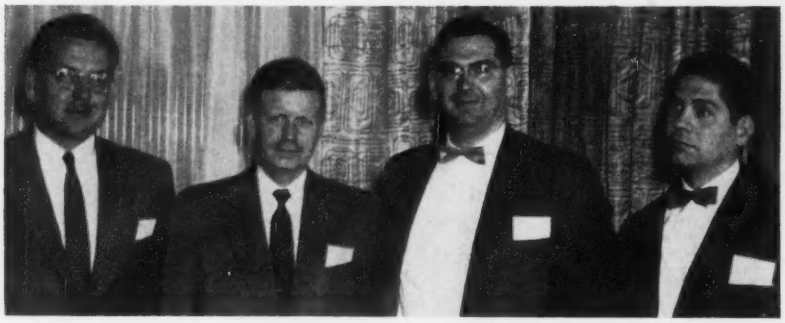
Moore to Join Committee

Dwight G. Moore has been appointed to the Editorial Review Subcommittee, according to Fred M. Reinhart, subcommittee chairman.

Mr. Moore will review technical papers on ceramic coatings being considered for publication in CORROSION.

He is affiliated with the Enameled Metals Section of Mineral Products Division at the National Bureau of Standards in Washington, D. C.

Northeast Region News



METROPOLİTAN NEW YORK SECTION Officers for 1959 installed at a recent meeting are shown left to right above: Secretary-Treasurer S. N. Palica, Consolidated Edison Co., Chairman A. F. Minor, American Telephone and Telegraph Co., Vice Chairman B. C. Lattin, Ebasco Services, and R. E. Gackenbach, member-at-large on Board of Governors who is affiliated with American Cyanamid Co.

Kanawha Valley Section will hold its March 26 meeting in Huntington, W. Va. Technical topic will be on underground coatings.

Ken Bloom of Armco Steel spoke on stress corrosion cracking of stainless steels at the January meeting.

●

Genesee Valley Section held a panel discussion on miracle water conditioners at its January meeting. Panel members were Gil Cox, International Nickel, Rochester, N. Y., R. N. Stenerson, Carrier Corp., Syracuse, R. Shaffer, Hall Laboratories, Pittsburgh, Pa., and Dave Priest, Pfaudler-Permutit, Rochester, N. Y.

●

Niagara Frontier Section elected 1959 officers at its joint meeting in January with the American Society of Metals. They are Chairman M. Stern, Electro Metallurgical Co., Niagara Falls, N. Y., Vice Chairman E. K. Benson, New York Telephone Co., Buffalo, and Secretary-Treasurer C. R. Bishop, Electro Metallurgical Co., Niagara Falls, N. Y.

Philadelphia Section February meeting included a talk entitled "A Corrosion Engineer Looks at Protective Coatings" by Ed Brink of American Viscose Corporation and also a member of the NACE Board of Directors.

●

Metropolitan New York Section heard John J. Moran, Jr., International Nickel Co., speak on high temperature corrosion at its February meeting.

Next meeting will be April 8 at the Military Park Hotel, Newark, N. J. A discussion on the various types of rectifiers will be held.

●

Southern New England Section scheduled John Wagner, Jr., of A. V. Smith Engineering Co., as speaker on concepts and applications of cathodic protetction at its February meeting.

●

Greater Boston Section held its 30th meeting in January with 31 members and 17 guests present. G. Carl Vogelsang of Gates Engineering Company spoke on elastomeric coatings for corrosion protection.

●

Baltimore-Washington Section will have a film and speaker on corrosion problems in nuclear reactors at its April 14 meeting.

W. J. Schwerdtfeger spoke on cathodic protection at the February meeting. A panel to discuss general questions on cathodic protection was also held. Panel members included the speaker, J. W. McAmis and H. S. Preiser.

●

Northeast Region Meeting will be held October 5-7, 1959, at Baltimore, Maryland.

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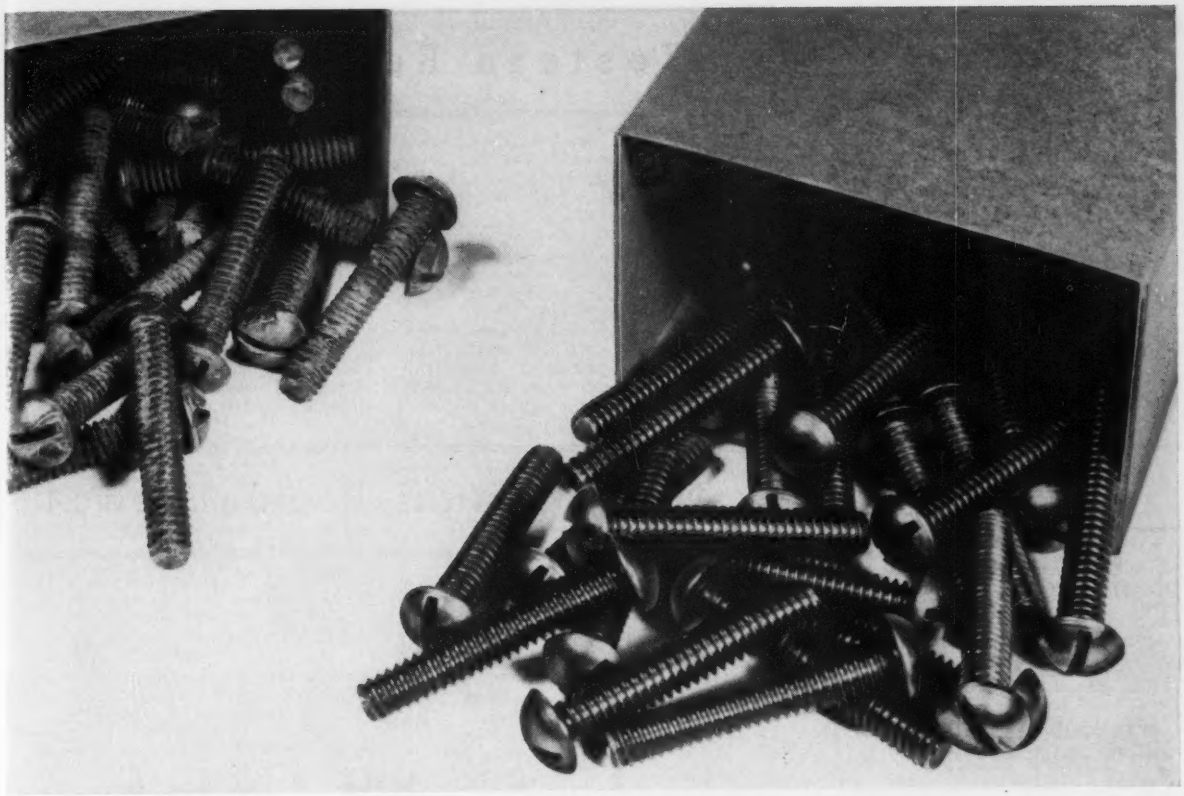

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BOOTH 125

National Association of Corrosion Engineers Show

Chicago March 16-20

Electrical Mechanical Division

GOOD-ALL ELECTRIC MFG. CO.
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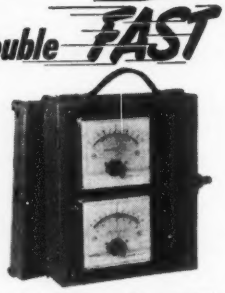
Locates Trouble FAST



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Oak Case, 9"x10"x4½"—Wt. 6 pounds.

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Western Region News

Central Arizona Section had Plastic Coated Steel Pipe as its meeting topic for January. H. H. McDaniels of Republic Steel Corporation was speaker.

•

Los Angeles Section meeting for January included a talk on application of the Flexiliner idea to corrosive storage facilities given by Wayne Jones.

•

Portland Section had the topics plastic piping and boiler corrosion at its January and February meetings. Sam Grub of Portco Corporation spoke on new developments in plastic piping in January; Robert S. Baynham, Dearborn Chemical Co., spoke at the February meeting on boiler corrosion.

•

San Francisco Bay Area Section meeting will be held March 11 at the Iron Duke in San Francisco. Ernest F. Ehmke will discuss recent corrosion problems at a fluid cat cracker and gas plant.

Prevention of scale and corrosion in cooling water was discussed by Ray W. Hawksley, consulting engineer, at the February meeting.

North Central Region News

Detroit Section will have NACE Vice President Hugh P. Godard as guest speaker at the March 20 dinner meeting.

Section officers for 1959 are Chairman Lewis Gleekman, Wyandotte Chemical Corp., Vice Chairman Euel Vines, Koppers Co., Treasurer Carl Curbin, Chrysler Corp., and Secretary Leonard Rowe, General Motors Corp.

The January meeting featured a talk on the effects of water quality on the corrosion of metals in Glycol antifreeze given by M. A. Boehmer.

David Roller, NACE member affiliated with the Wright Air Development Center spoke on thin metal film corrosion indicators at the section's joint meeting with the Electrochemical Society in February.

•

Greater St. Louis Section officers for 1959 are Chairman Oliver W. Siebert, Vice Chairman Walter B. Meyer, Secretary E. G. Johannes, Treasurer David E. Morris and Board Members Larry Goad, Otto H. Fenner, W. C. Linton and J. Vandeventer.

•

Kansas City Section members saw the film "Melting of Special Steels" at its January meeting.

Committee assignments were made: R. M. Fuller, membership; Charles E. Gillam, attendance; and Orville E. Moore and Fred Few, program.

•

Southwestern Ohio Section had a review of the proper use of plastic pipe and lined pipe given by Herbert D. Stuber, Saran Lined Pipe Co., at its January meeting.

Canadian Region News

Regional Officers for 1959 have been elected. They are Regional Director T. R. B. Watson, Corrosion Service Ltd., Toronto; Chairman H. G. Burbidge, Aluminum Co. of Canada Ltd., Montreal; Vice Chairman J. R. Grey, B. C. Electric Co., Ltd., Burnaby, B. C.; Secretary-Treasurer H. Yates, McGill University, Montreal; and Membership Chairman F. W. Hewes, Canadian Protective Coating Ltd., Edmonton, Alberta.

DEATHS

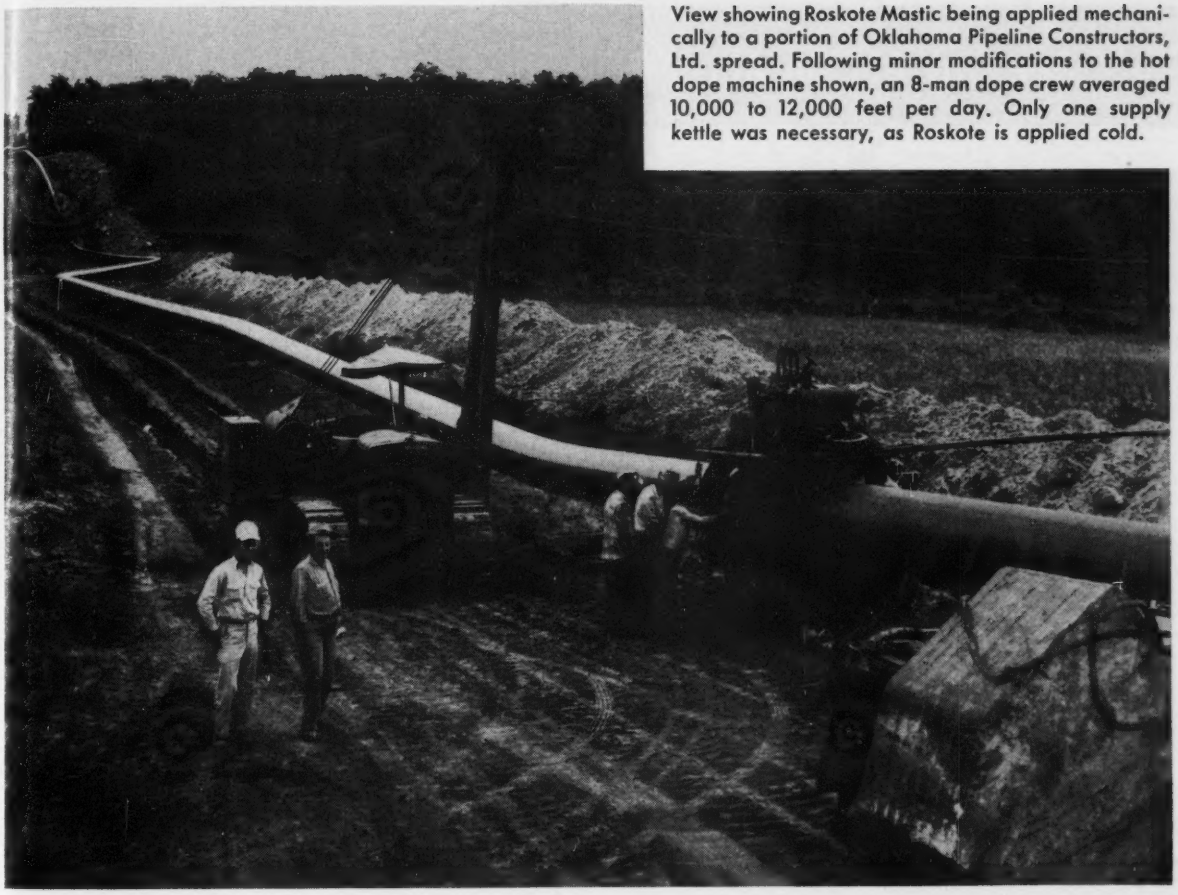
John C. Watts, Jr., NACE member and engineering editor of Gulf Publishing Company's Pipe Line Industry Magazine, died February 8. Formerly, he was corrosion engineer with Humble Pipe Line Company. He was a graduate chemical engineer from Mississippi State College and held memberships in the Houston Pipe Liners Club and the Houston Engineering and Scientific Society.

Publication Procedure For Conference Papers

Technical papers presented at meetings of the National Association of Corrosion Engineers, will, after review and approval by the NACE Editorial Review Subcommittee and the editor of CORROSION be published in the Technical Section of the magazine. Persons interested in the material thereby will have access to the information in CORROSION, which is extensively indexed both by NACE itself and by other agencies.

Corrosion workers concerned with technical literature originating with and published by NACE are invited to write to the association for copies of the NACE Literature List.

The association does not publish a proceedings of its meetings. Publication of technical papers usually follows their presentation by periods of months, because of the time consumed in review, editing and revision. The association also considers for publication technical articles submitted without invitation on any phase of the corrosion control problem.



View showing Roskote Mastic being applied mechanically to a portion of Oklahoma Pipeline Constructors, Ltd. spread. Following minor modifications to the hot dope machine shown, an 8-man dope crew averaged 10,000 to 12,000 feet per day. Only one supply kettle was necessary, as Roskote is applied cold.

Roskote Applied Cold with Line Travel Equipment on Trans-Canada 30 Inch

Over rough terrain, with a right of way that crossed solid granite, muskeg, swamp and glacial till, and working under adverse weather conditions, the world's longest and most difficult pipeline construction — Trans-Canada Pipe Lines, Ltd. — has been completed.

One contributing factor in the success of this construction was the adaptation of standard line travel dope machines for the easy application of Roskote cold mastic. This combination of Roskote Mastic and modified line travel machines made possible the coating of ten to twelve thousand feet per day of 30-inch piping on the downstream side of the compressor stations.

Contractors* were able to carry on the coating operation with an 8-man dope crew, using only one supply kettle, requiring no heating fuel and thereby eliminating toxic fumes and fire hazard.

*Contractors on Trans-Canada who applied Roskote by line travel equipment:
Morrison-Shivers, Ltd., Majestic Contractors, Ltd.,
B. C. Rivers Construction, Ltd., Dutton-Williams Brothers, Ltd.
and Oklahoma Pipeline Constructors, Ltd.

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Please send me complete information about the economics and application of cold-applied Roskote Mastic with line travel equipment.

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Company.....

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City..... Zone..... State.....

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Requirement: Low-cost, corrosion-resistant heat exchanger material
Key to Good Design: Specify Alcoa Aluminum Tube and Sheet

If corrosion resistance, long life, product purity and economy are considerations in your heat exchanger application, specify aluminum! ALCOA® Aluminum has been solving such problems for over 40 years.

In the petroleum industry, ALCOA Aluminum Tubes serve in main column overhead condensers where their freedom from fouling and excellent heat transfer properties have made them a natural selection. Heat exchangers made of ALCOA Aluminum Tube and Plate are used in the production of ammonia, nitric acid, vegetable oils, naval stores, naphthalene, hydrogen peroxide, oxygen and urea. These and other processes call for heat exchangers with combinations of high strength, excellent sub-zero physical qualities, high thermal conductivity, or nontoxic and noncatalytic properties. ALCOA Aluminum alloys meet all these requirements.

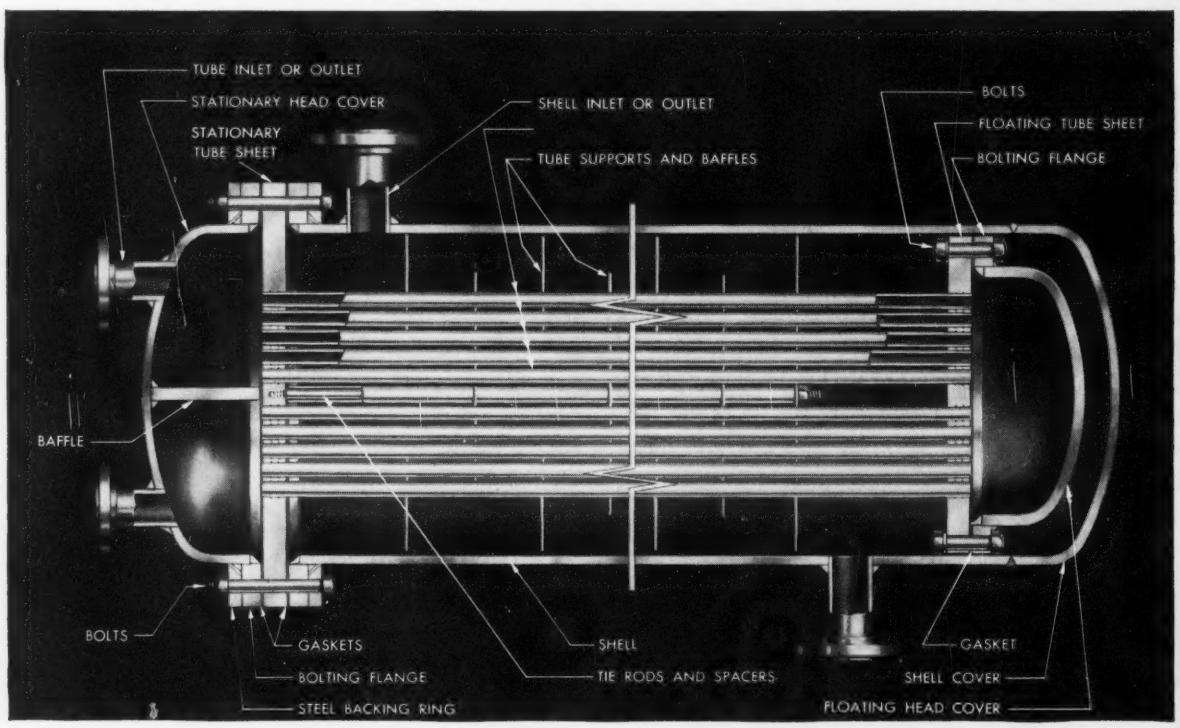
As for cost, ALCOA Aluminum Tube is the most inexpensive. In the common sizes it costs one-third less than mild steel, one-half as much as admiralty, and one-fifth that of stainless.

ALCOA Aluminum alloys are ideally suited to a wide range of maintenance methods. For relatively soft or loose scale, water jets and brushes are usually satisfactory. Wet sandblasting as well as chemical cleaners may be used effectively.

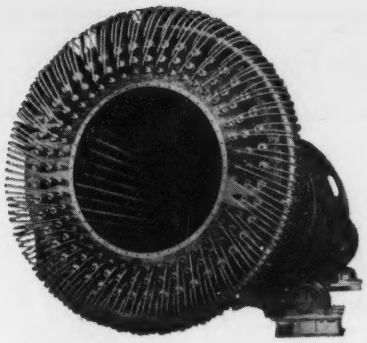
ALCOA engineers have worked closely with all segments of the process industries for over 40 years, and can help you specify the aluminum alloy best suited to your heat exchanger and other process applications. ALCOA's unparalleled experience in this field is available to you for the asking. Write to the address on the coupon, stating your requirements as specifically as possible. ALCOA's development engineers will welcome the opportunity to work with you on your problems.

You can also take advantage of the wide selection of free ALCOA literature on aluminum for heat exchangers and other process applications. Simply check the booklets you want on the coupon and mail to the address indicated. ALCOA will forward your material promptly and without obligation.

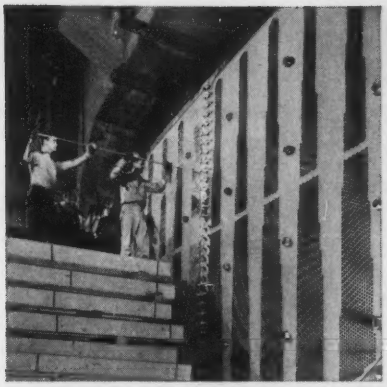
ALCOA is conducting a series of engineering conferences on process industries applications of aluminum during 1959 in a number of major cities. Contact your nearest ALCOA sales office for full particulars and dates.



For certain processes where corrosive or sensitive materials are involved, it is often advisable to use aluminum for entire heat exchanger systems to avoid system corrosion and contamination of the product. A typical all-aluminum heat exchanger is shown here. Alloy recommendations are given in the table opposite.

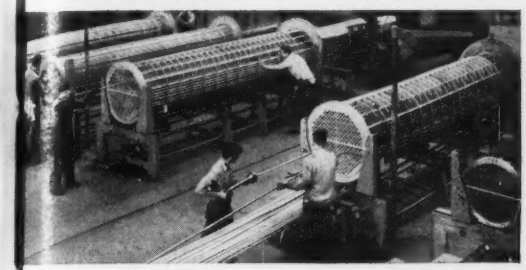


Alcoa Aluminum Tubing protects color in the world's largest rotary steam-tube drier. The drier measures 10 ft in diameter by 100 ft long.

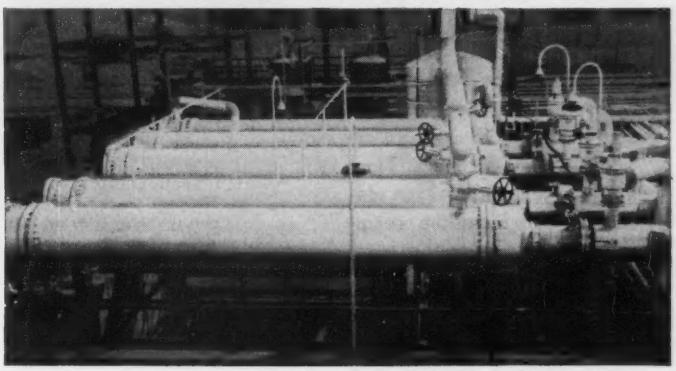


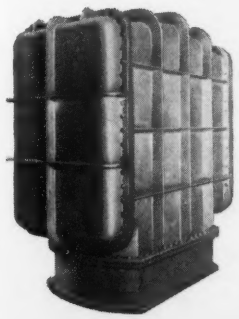
A Midwest utility saved 50 per cent on tubing costs by using Alcoa Aluminum instead of admiralty for tubing in this surface condenser. Successful operation of this unit has led to the installation of similar units by other utilities. This use demonstrates aluminum's economy and superior resistance to ammonia, carbon dioxide and hydrogen sulfide.

A battery of all-aluminum heat exchangers in a polyethylene plant. Selected for initial low cost, these aluminum exchangers provide freedom from product contamination as well as desirable low temperature properties.



Construction of a heat exchanger utilizing Alcoa Aluminum Tube. Superior working properties plus the cold-drawing method by which Alcoa seamless heat exchanger tubes are produced results in close dimensional tolerances and smooth inside and outside surfaces.





This all-aluminum dephlegmator in a large coke plant is used for the regeneration of ammonia liquor in a hydrogen sulfide scrubbing operation. Aluminum tubes lasted five times longer than steel tubes in a similar use.

Alloy Recommendations for Aluminum Heat Exchanger Construction

	Alcoa Designation	ASTM Specification*	Alloy
Tubes	3003-H14	B234	M1A
	Alclad (inside) 3003-H14	B234	clad M1A
	Alclad (outside) 3003-H14		clad M1A
	Alclad (both sides) 3003-H14		clad M1A
	6061-T6	B234	GS11A
	6062-T6	B234	GS11C
Tube Sheets	3004-H112	B178	MG11A
	Alclad 3004-H112	B178	clad MG11A
	6061-T6	B178	GS11A
	Alclad 6061-T6	B178	clad GS11A
Baffles	3003-H14	B178	M1A
	Alclad 3003-H14	B178	clad M1A
Tie Rods	6061-T6	B273	GS11A
Shells & Headers	3003-H112	B178	M1A
	Alclad 3003-H112	B178	clad M1A
	3004-H112	B178	MG11A
	5052-H112	B178	GR20A
	5154-H112	B178	GR40A
Fittings	3003-F	B274	M1A as applicable
	6061-T6	B274	GS11A as applicable
Flanges	6061-T6	B247	GS11A as applicable

*Only the basic number is shown since Alcoa products are manufactured to the latest issue of the specification. The complete number would indicate the year of issue.

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Please send me the following literature covering Alcoa Aluminum in heat exchanger and other applications in the process industries.

- ☐ 10186 Alcoa Aluminum Heat Exchanger Tubes
- ☐ 20437 Aluminum Alloy Heat Exchangers in the Process Industries
- ☐ 10460 Process Industries Applications of Alcoa Aluminum
- ☐ 20849 Resistance of Aluminum Alloys to Weathering and Resistance of Aluminum Alloys to Chemically Contaminated Atmospheres
- ☐ 20272 Aluminum Alloys for Handling High Purity Water
- ☐ 20268 Resistance of Aluminum Alloys to Fresh Waters
- ☐ 20935 Designing to Prevent Corrosion

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Wayne A. Johnson, president, and his associates in Corrosion Rectifying Co., Inc., are well-known corrosion prevention specialists with headquarters in Houston and Southeastern Divisional offices in New Orleans. Their work is concentrated on designing and installing corrosion rectifying systems for pipelines, refineries and oil producing companies. Call on them for prompt and experienced surveys, systems and materials.

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- **FEASIBILITY STUDIES**
- **DESIGNS**
- **INSTALLATIONS**
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President

JAMES T. CONDRY
Southeastern
Division Manager

CORROSION RECTIFYING CO., INC.
5310 Ashbrook MO 7-6659
P. O. Box 19177 HOUSTON, TEXAS

South Central Region News

NORTH TEXAS SECTION officers for 1959 are shown above, left to right: E. C. Rogness, telephone committee chairman, Secretary-Treasurer Jerry McIntyre, Chairman R. B. Bender, Vice Chairman Don Taylor and Section Trustee Glyn Beesly.

Alamo Section saw a film titled "Life Time Protection for Steels" at its January meeting. The film was presented by Dave Whiteman, Amercoat Corp.

•

Houston Section held its annual students' night meeting last month. About 50 students from Lamar Tech, University of Houston, Rice Institute, Texas A & M and University of Texas were invited. Wayne Friend of International Nickel Co., Inc., spoke on the properties of high strength, high temperature alloys.

W. W. Akers, Rice Institute chemistry professor, gave a general appraisal of the future course of engineering at the January meeting.

An Education Committee for the section has been appointed. Committee chairman is John Loeffler. Other members are C. B. Tinsley, M. A. Riordan, Walter Noser, C. L. Woody, George Hall, John L. Weis, Mel Judah, Oscar Hunter and Robert Anderson. This committee also will cooperate in the education program of the Engineers Council of Houston.

•

Greater Baton Rouge Area Section members will hear Phil Wogan speak on coatings and paints at the March 23 meeting to be held at the Bellemont Motor Hotel in Baton Rouge.

New officers are Chairman Arthur H. Tuthill, Valco Engineering, Inc., Vice Chairman Donald C. Townsend, Ethyl Corp., and Secretary-Treasurer W. E. Doescher, Jr., Levingston Supply Co. Trustee R. M. Eells, Esso Standard Oil, will finish his term in 1959.

Corrosion in chemical and petrochemical plants by John Mason and coatings were the topics for the January and February meetings.

•

North Texas Section heard William H. Wade of the University of Texas speak on radiation effects of corrosion at its February meeting.

•

Corpus Christi Section officers for 1959 are Chairman William R. Taylor, Vice Chairman Fred C. Reeb, Secretary-Treasurer Leon Lankford and Trustee Paul Laudadio.

A panel discussion was held at the section's January meeting.

Teche Section 1959 officers were installed at a January dinner-dance meeting. They are Chairman John D. Stone, Sunray Mid-Continent, Vice Chairman Sam F. Fairchild, Sunray Mid-Continent, Secretary Ron J. Cernik, Union Oil of California, Treasurer Phil C. Brock, Dowell, Inc., and Trustee O. L. Bassham, Tret-o-lite Co.

The section's meetings for 1959 are scheduled for March 26, April 30, May 21, June 3 (Short Course Banquet), June 18, July 30, August 27, September 24, October 22, November 19 and December 3.

•

Rocky Mountain Section officers recently elected are Chairman Herbert L. Goodrich, American Telephone & Telegraph, Vice Chairman Burton K. Wheatlake, B. K. Wheatlake Co., Secretary-Treasurer William P. McKinnell, Jr., Ohio Oil Co., and Trustee John R. Hopkins, Protector Wrap Co.

•

Permian Basin Section heard Floyd Blount of Magnolia Petroleum's Research Laboratory speak on non-destructive testing of oil field materials at the February meeting.

Jim Greedy and Chuck Davin of Pipe Linings, Inc., presented films and spoke on in-place cement lining of pipe at the section's January meeting.

•

West Kansas Section will elect a new secretary-treasurer. J. J. Crockett, who was recently elected to that office, has resigned.

•

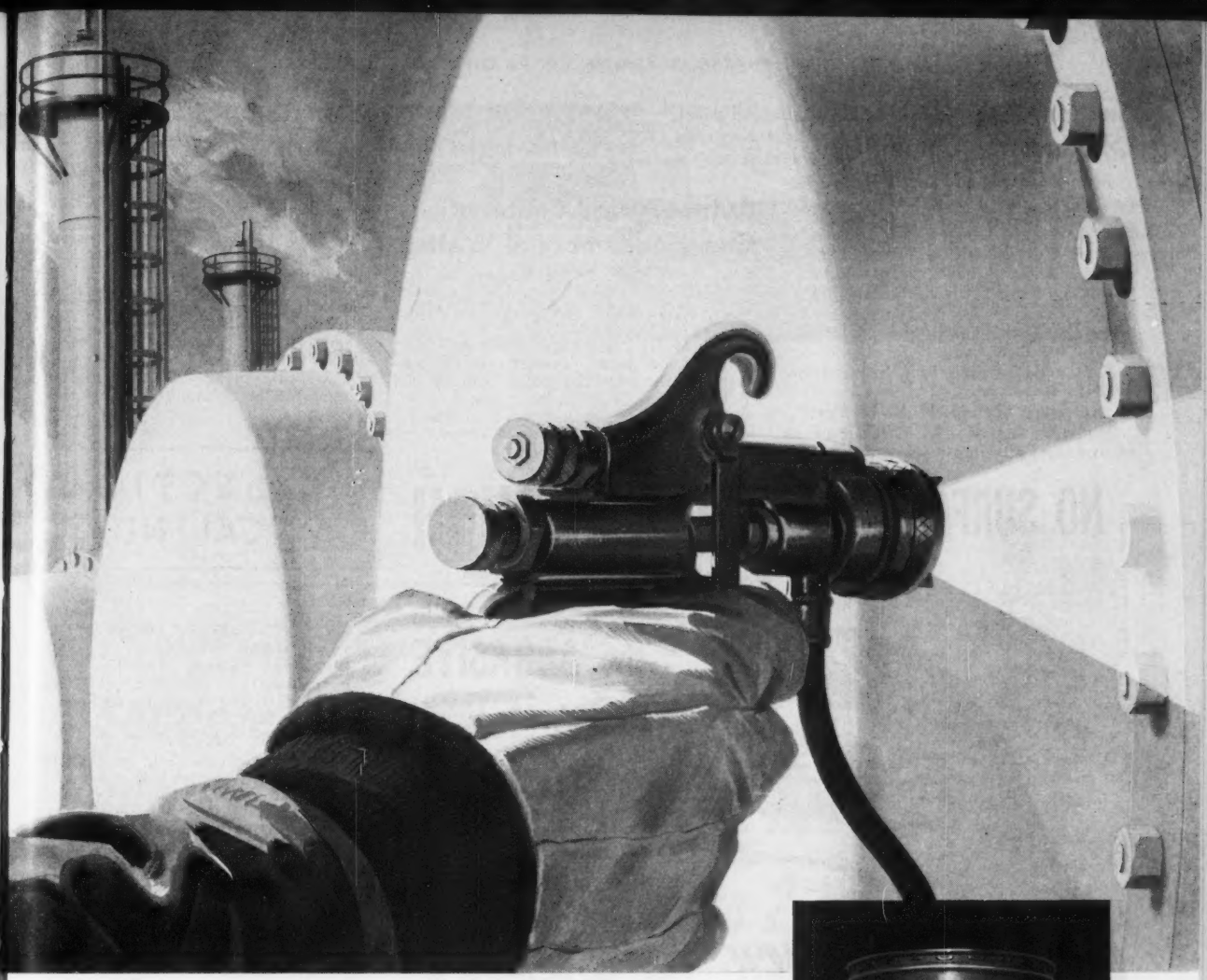
East Texas Section held a buffet dinner for its January meeting with Maurice Belson, C. D. Stearnes Co., Shreveport, as the guest speaker on high voltage inspection of protective coatings.

•

Shreveport Section February meeting included a talk by Robert M. Nee of Nee and McNaulty, Inc., Tulsa, on underground pipeline coatings and tape coatings.

At its January meeting, the section heard Bob Sinclair of United Gas Pipeline, Carthage, Texas, speak on corrosion problems in gasoline plants.

(South Central Region News Continued on Page 94)



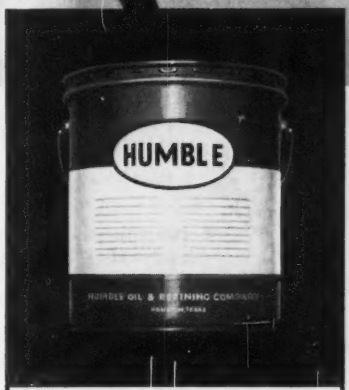
Humble Coatings Give You Three-Way Reduction in Maintenance Costs!

Humble coatings last six months to a year longer than most other paints. You save *two* ways — on paint and labor. The *third,* and most important, way you save is this: you get better protection, which means longer life for expensive equipment.

Humble coatings contain rust-preventing ingredients that not only provide surface protection, but also neutralize any corrosive elements that may penetrate the surface protection and attack the metal. These coatings were specially developed to give maximum protection in refineries and chemical plants where strong acids, alkalis and salt sprays produce severe corrosive conditions.

Without cost or obligation, one of Humble's trained engineers will make a thorough study of your needs and recommend a complete painting program for you. Call your nearest Humble representative, or phone or write:

HUMBLE OIL & REFINING COMPANY
Consumer Sales, P.O. Box 2180,
Houston 1, Texas



Humble's line of coatings includes —

- RUST-BAN ®
- Epoxies
- Phenolics
- Tank coatings
- Vinyls
- Acrylics
- Enamels
- Hot surface coatings
- Interior and exterior emulsion-type paints

HUMBLE

Sections Appoint Heads Of Education Committees

Five sections of the South Central Region have appointed Educational Committee Section Chairmen, according to John Loeffler, South Central Region Educational Committee Chairman. They are as follows:

Rocky Mountain Section: Herbert L. Goodrich.

East Texas Section: J. H. Graves, Tidal Pipe Line Co.

Panhandle Section: John F. Headrick, Phillips Petroleum Co.

Shreveport Section: H. V. Beesley.

Houston Section: J. E. Loeffler as chairman of a committee to include C. B. Tinsley, M. A. Riardan, Walter Noser, G. L. Woody, George Hall, John L. Weis, Mel Judah, Oscar Hunter and Robert Anderson.

Author Award Contribution Given in Memory of Watts

A contribution to the Young Author Award Fund has been made in memory of John C. Watts, Jr., NACE member who died February 8.

The award was established in 1953 by an anonymous donor to recognize authors of meritorious papers published in CORROSION. It is given to the author who is under 30 years of age at the time his paper is submitted to CORROSION for publication.

Baton Rouge Section Gives Industrial Painters Course

A training course for industrial painters is being sponsored by the Baton Rouge Section. Classes began February 9 and will continue through March 23. Meetings are held each Monday night from 7 to 9 pm.

A special class consisting of practical demonstrations will be given on Saturday, March 28, when certificates will be presented to those having satisfactorily completed the course.

Classes are being held at the Baton Rouge Trade School on Winbourne Avenue.

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BLACK BEAUTY ▶ BLAST GRIT

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★ Fastest cutting, sharpest material known

★ Longer lasting, reuseable many times over

★ Moisture-free no packing from dampness. FREE FLOWING.

★ Leaves surfaces absolutely clean—no deposits

★ Gives highest degree of etch for better bonding.

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SECTION CALENDAR

Mar.

3 Shreveport Section. Caddo Hotel.
5 West Kansas Section.
5 Pittsburgh Section. Activities of the Prevention of Deterioration Center at the National Academy of Science, by C. J. Wessel.
6 Birmingham Section.
9 Central Oklahoma Section. Tropical Cafeteria, Oklahoma City.
10 Montreal Section. Corrosion Work in the National Research Council, by Morris Cohen.
11 Greater Boston Section. Corrosion Behavior and Protective Value of Electrodeposited Coatings, by Clarence H. Sample, Inco.
11 San Francisco Bay Area Section. The Iron Duke in San Francisco. Corrosion Problems at a Fluid Cat Cracker and Gas Plant.
20 Detroit Section. Hugh P. Godard, NACE President, speaker.
23 Greater Baton Rouge Area Section. Bellemont Motor Hotel, Baton Rouge. Talk on coatings and paints by Phil Wogan.
26 Teche Section. Petroleum Club.
26 Kanawha Valley Section. Huntington, W. Va. Underground Coatings.
30 Atlanta Section.
31 Panhandle Section.

April

2 West Kansas Section.
6 North Texas Section.
7 Shreveport Section. Caddo Hotel.
8 Metropolitan New York Section. Military Park Hotel, Newark, N. J. Discussion of various types of rectifiers.
14 Baltimore-Washington Section. Film and speaker on corrosion problems in nuclear reactors.
21 Chicago Section. How to Paint Tanks Economically, by K. R. Walston, Standard Oil Co. (Ind.).
21 Cleveland Section. Cleveland Engineering and Scientific Center. Protective Coatings, by Stanley Lopata.
28 Panhandle Section.
30 Teche Section. Petroleum Club.

•

The University of Oklahoma Corrosion Control Short Course will be held March 31-April 12 at the North Campus, Norman.

Well	BEFORE TREATMENT		AFTER KONTOL SQUEEZE TREATMENT	
	Tubing Failures Per Year	Iron Loss (Lbs/Day)	Tubing Failures Per Year	Iron Loss (Lbs/Day)
A	2	3.8	0	0.7
B	2	4.5	0	0.7
C	4	8.8	2	4.3
D	1	10.1	0	0.6
E	1.7	11.2	0	1.6
F	2	8.8	0	0.5
G	1.3	4.7	0	1.0
H	1.5	3.3	0	0.5
	15.5	55.4	2	9.9

See our exhibit at the International Petroleum Exposition, May 14-23, and 5th World Petroleum Congress, June 1-5.

another
TRETOLITE SERVICE
report

87% reduction
saves $52,000 annually

Squeeze Application of KONTOL*CORROSION INHIBITOR REDUCES TUBING FAILURES FROM 15.5 to 2 PER YEAR

A corrosion control program employing KONTOL substantially paid off on a Texas lease. Based on the cost of pulling wells and replacing corroded tubing, an average of $4,000 per job, the savings in one year (based on figures in table above) approximated $52,000.

HOW KONTOL PROTECTION WAS ACHIEVED

KONTOL Corrosion Inhibitor was squeezed into the well formations at the rate of 55 gallons per well, followed by 2 quarts of TRET-O-LITE* demulsifying chemical in 15 barrels of oil, followed by oil injection sufficient to displace the liquid in the tubing and force the chemical into the formation. Injection was obtained by means of a 5 g.p.m. triplex pump and 40 g.p.m. centrifugal pump.

COST OF TREATMENT

The KONTOL squeeze treatment cost about 70 mills per barrel of produced oil and 1.9 mills per barrel of fluid produced. This resulted in a treating cost of 17 mills per barrel less than previous corrosion prevention methods.

*KONTOL *and* TRET-O-LITE *are registered trade-marks of Petrolite Corp.*

This is just one of many cost savings stories on KONTOL Corrosion Inhibitor treatment. For more —and information on how to start a KONTOL Corrosion Control Program—ask the Man in the Red Car. Or write to . . .

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MEXICO: R. E. Power, Sierra de Mijes, No. 125, Mexico, D. F.
NETHERLANDS: F. E. C. Jenkins, Hoefbladlaan 134, The Hague
PERU: International Gas Lift Company, Apartado 71, Talara
TRINIDAD: Neal and Massy, Ltd., Port of Spain, P.O. Box 544, San Fernando

10 Years of NACE Educational Activities

72 Corrosion Control Short Courses Given

Recognizing the need to supplement the professional training of engineers working in the field of corrosion control, the National Association of Corrosion Engineers has sponsored, co-sponsored or participated in 72 corrosion control short courses during the past 10 years. Many of the courses have been organized to be useful to field and operating personnel.

Because few colleges or universities offered regular curriculum courses in corrosion, most engineers going into corrosion control work have realized the need for additional training.

25 Colleges Participate

Twenty-five colleges and universities have participated in these short courses. The institutions were in 16 states, 2 provinces of Canada and in Mexico. Many of the colleges and universities which have participated in the NACE short courses have added regular course work on corrosion to their curricula.*

The first NACE corrosion short course was held at the University of Texas in 1949. Since then, several courses have been established as annual events. Tulsa Section of NACE has held courses annually for ten years; Shreveport Section has held its eighth annual course. Both will hold courses during 1959.

Oklahoma University and the Central Oklahoma Section have sponsored five annual short courses and will hold their sixth this year. The University of Illinois will hold its fourth biennial short course in 1959.

Subject Matter Taught

Subjects covered at courses during the past ten years are diverse. Most frequent were fundamentals of corrosion, fundamentals of cathodic protection, coatings and linings, corrosion inspection, surveys and tests, and inhibitors.

Economic and management problems of corrosion control as an area of instruction and the study of structural design for corrosion control have been taught more often in recent years.

The trend in subject matter during the past ten years has been from the general to specialized subjects. Biological factors of corrosion, surface treatment and selection of structural materials are among specialized subject taught at recent courses.

Early short courses considered only the fundamentals of cathodic protection, but recently such specialized areas as potential measurements, instruments, holiday detection, rectifiers, galvanic anodes, backfill, coatings, wrappings, system design and power distribution systems have been taught.

Several short courses have been designed to train personnel in particular industries such as the Tulsa Section's and the Houston Section's short courses designed primarily for men in the pipeline industry. Other short courses have been specialized in a particular subject area such as the Appalachian short course on underground corrosion held at the University of West Virginia.

Specialized Short Courses

Other specialized short courses were those held at the University of North Dakota on maintenance coatings and at Ohio State University on the metallurgical aspects of corrosion. A short course held at the University of California at La Jolla was on marine corrosion. Other short courses considered corrosion control from the viewpoint of a particular industry such as the chemical industry.

Most of the short courses are presented on one or more of three levels of instruction to satisfy the needs of industrial personnel involved in corrosion control work. The courses may be aimed primarily at engineers, foremen and supervisors, or at persons less directly concerned with corrosion control but who wish to be familiar with the subject. This latter level of instruction appeals to inspectors, field men and sales and service personnel besides serving also as a refresher.

Methods of instruction include lectures, discussions, panels and demonstrations. Average length of the courses has been 3½ days. Some courses have been one-day seminars; others have lasted five days. One college held 15 weekly sessions at night to accommodate professional engineers in its local area.

Average attendance during the past ten years has been about 147 per course.

Engineers and Professors Teach

Faculties for the courses have consisted mostly of experienced engineers involved in corrosion control work. When the short course is held in cooperation with a college or university, the faculty of the institution usually instructs some classes also. A majority of the engineers who teach at the short courses and many of the college faculty are members of NACE.

Seven corrosion control short courses have been scheduled for 1959, including those of Kansas City Section at St. Teresa College, Houston Section at the University of Houston and Tulsa Section. These three courses were held earlier this year. Central Oklahoma Section's short course at the University of Oklahoma will begin March 31.

Other short courses for 1959 include Shreveport Section's at Centenary College, Teche Section's at Southwestern Louisiana Institute and the Appalachian Underground Corrosion Short Course at the University of West Virginia.

Dates for short courses are regularly given in the National Regional Meetings and Short Courses calendar each month in CORROSION.

Other instructional activities in which NACE has participated are special corrosion tours such as the annual Permian Basin Corrosion Tour sponsored by the Permian Basin Section in Texas and the San Joaquin Valley Corrosion Tour in California. These tours offer engineers an opportunity to see field exhibits of corrosion on equipment and materials and corrosion control methods used by various companies and to discuss remedial measures.

Another NACE educational activity includes the nine-day corrosion seminar held at Louisiana State University in 1955.

* For additional information on college courses on corrosion, see R. V. James article entitled "Corrosion Control Courses—What Colleges Are Teaching," Page 122, Corrosion, December, 1958.

Copies of Papers

Copies of papers presented at the 15th Annual NACE Conference at Chicago can be obtained by registrants only. A procedure has been arranged whereby

1. Persons who register at the conference may order copies of the papers in which they are interested.

2. Orders must be placed at the conference registration desk during the conference.

3. A small charge, sufficient to reimburse NACE for the cost of preparing photographic copies will be made for each paper ordered.

4. Papers ordered will be copied after the conference is over and mailed.

T. J. Hull, NACE executive secretary, said the association will not accept orders for copies from persons who are not registered at the conference.

Manuals, Films and Research Included in NACE Activities

The National Association of Corrosion Engineers has participated in other educational and training activities in addition to fostering corrosion control short courses. These activities have included publication of instructional manuals, scripts for motion picture films and research projects.

Motion Pictures

The NACE Advisory Committee on Motion Picture Films is currently working with the University of Texas Petroleum Extension Service on the scripts of a three-part film series entitled "Soil Corrosion of Pipe Lines."

Instructional Manual

NACE Technical Committee T-1 on Corrosion in Oil and Gas Well Equipment co-sponsored with the American Petroleum Institute the preparation of a manual entitled "Corrosion of Oil and Gas Well Equipment," published last September. The manual was designed to give field operating personnel an understanding of the enormous cost of corrosion, to help them recognize indications of corrosion and to realize the importance of early detection.

Research Program

Technical Committee T-1G on Sulfide Stress Corrosion Cracking supervised a four-year research program sponsored by industry and Yale University on hydrogen absorption, embrittlement and fracture of steel. Results of the research were published as a committee report in the July, 1957, issue of CORROSION.

Short Course Planning

Technical Committee T-1 is also working with the American Petroleum Institute to help plan short courses on the corrosion of oil and gas well equipment. The courses are to be sponsored by the API.

High School Awards

The South Central Region of NACE has given cash awards to high school students who presented the best corrosion displays at the 1957 and 1958 Oklahoma State Fairs.

Manuals For Science Teachers

The Houston Section has appointed a committee to prepare manuals on the fundamentals of corrosion for use by high school science teachers to stimulate more interest in science study in the public schools.

Corrosion Control Short Courses for 1959

Oklahoma University Short Course Set For March 31-April 2

Subjects and speakers have been scheduled for the Corrosion Control Short Course to be held March 31-April 2 at the Extension Study Center, University of Oklahoma, Norman.

Co-sponsored by the University and Central Oklahoma Section, the short course is designed for persons in corrosion control program who are not experts in the field.

Subjects to be presented are scheduled as follows:

Tuesday, March 31

Economics of Corrosion Control
Fundamental Concepts of Corrosion
Fundamentals of Cathodic Protection
Fundamentals of Corrosion Control by Inhibitors
Sand Box Demonstration

Wednesday, April 1

Corrosion Problems in Water Flood Projects
Protecting Surface Equipment
Protecting Subsurface Equipment Inside Oil Well Casing
Protecting Oil Well Casing
Cathodic Protection of Pipelines
External Coatings for Pipelines
Making Cathodic Protection Work

Thursday, April 2

Corrosion Resistant Materials
Effect of High Pressure and High Temperature on Various Hydrochloric Acid Inhibitor Systems
Control of Wood Deterioration in Cooling Towers
Water Treatment for Injection Into Wells
Meaning and Use of Water Analysis
Importance of Recognizing and Reporting Corrosion Damage by Maintenance Foremen
Round Table Discussion

Speakers

Speakers scheduled to participate in the course include J. C. Spalding, Jr., Sun Oil Co., O. W. Everett, Oklahoma Natural Gas, W. C. Koger, Cities Service, B. D. Oakes, Dowell, Inc., Tom Blenkinsop, Betz Laboratories, Don Oleen, Nalco, R. R. Rothrock, D-X Sunray Oil Co., and Lyle Sheppard, Shell Pipeline Co.

Houston Short Course

An increase of 64 percent in attendance over last year was experienced at Houston Section's 5th Annual Corrosion Control Short Course held January 22-23 at the University of Houston.

Six states were represented by the 135 registrants in comparison with five states and 86 registrants at the 1958 short course.

Foremen, electricians and technicians, chemists, engineers, salesmen and management personnel were represented.

Several new NACE memberships were obtained during the course, according to W. P. Noser, chairman of the planning committee.

NATIONAL and REGIONAL MEETINGS and SHORT COURSES

1959

March 16-20—15th Annual Conference and 1959 Corrosion Show. Sherman Hotel, Chicago.

Sept. 29-30, Oct. 1—Western Region Conference. Bakersfield Inn, Bakersfield, Cal.

Oct. 1-2—Southeast Region. Jacksonville, Florida.

Oct. 5-7—Northeast Region. Lord Baltimore Hotel, Baltimore, Md.

Oct. 12-15—South Central Region Meeting, Denver, Col. Cosmopolitan Hotel.

Oct. 20-22—North Central Region, Cleveland.

1960

March 14-18—16th Annual Conference and 1960 Corrosion Show. Dallas, Texas, Memorial Auditorium.

Oct. 11-14—Northeast Region Meeting. Huntington, W. Va.

Oct. 19-20—North Central Region. Milwaukee.

Oct. 25-28—South Central Region Conference, Mayo Hotel, Tulsa.

1961

March—17th Annual Conference and 1961 Corrosion Show. Buffalo, N. Y., Hotel Statler.

Oct. 24-27—South Central Region Conference, Houston, Shamrock Hotel.

1962

March—18th Annual Conference and 1962 Corrosion Show. Kansas City, Municipal Auditorium.

October 16-19—South Central Region Conference, Hilton Hotel, San Antonio, Texas.

SHORT COURSES

1959

March 31-April 2—Corrosion Control Short Course. University of Oklahoma-Central Oklahoma Section. North Campus, University of Oklahoma, Norman.

June 2-4—Teche Section. Corrosion Control Short Course, Southwestern Louisiana Institute, Lafayette.

June 2-4—Appalachian Underground Corrosion Short Course, West Virginia University, Morgantown.

June 22-26—Massachusetts Institute of Technology Advanced Short Course in Fundamentals of Corrosion Reactions and Corrosion Control, Cambridge.

December 7-11—University of Illinois Corrosion Control Short Course. Urbana Campus.

•

An average of four to six short courses on corrosion control are held each year in the United States with the cooperation of NACE.

MIT Short Course On Advanced Level To Be June 22-26

An advanced short course in fundamentals of corrosion reactions and corrosion control will be given at Massachusetts Institute of Technology June 22-26.

The course will be designed for those who have had previous corrosion instruction as given at short courses sponsored by NACE or who have experience in the corrosion field and wish to enlarge their understanding of fundamentals.

Lectures will emphasize reaction mechanisms and interpretations of laboratory and field measurements from the standpoint of modern electrochemical and metallurgical concepts.

Subjects to be covered are the meaning and interpretation of electrode potentials and polarization measurements, calculation of corrosion rates from polarization data, passivity in metals and alloys, theory of cathodic protection and suitable criteria of complete protection, metallurgical factors affecting corrosion in particular stress corrosion cracking and corrosion fatigue, mechanisms of corrosion inhibitors and their proper application, and mechanisms of oxidation and tarnish.

Question and answer session on application of principles to practical situations will be held each day.

Acquaintance with elementary physical chemistry is suggested for those attending the course.

H. H. Uhlig, NACE member and director of MIT's Corrosion Laboratory, will be in charge of the short course.

Lecturers include Norman Hackerman, University of Texas, Bruce Chalmers, Harvard University, and Milton Stern, Metals Research Laboratories.

Inquiries should be addressed to James M. Austin, Director of Summer Session, Massachusetts Institute of Technology, Cambridge 39, Mass.

Appalachian Short Course Scheduled for June 2-4

Plans for the June 2-4 Appalachian Underground Corrosion Short Course include 64 classes and field demonstrations. The course will be held at West Virginia University, Morgantown.

Charles L. Dey of Koppers Co., Pittsburgh, Pa., is chairman of the General Committee.

Basic, intermediate and advanced education in four subject areas will be offered:

1. Fundamentals of underground metallic corrosion and corrosion control practices as related to underground pipe and cable systems.

2. Water: internal and external corrosion of metallic water systems and corrosion control measures.

3. Coatings: corrosion control materials and material control practices.

4. Special: instrumentation, materials and test procedures.

Final program for the course will be released later.

Letters to the Editor

Fuels Containing Boron

Wright Air Development Center
Area B, Box 8188
Wright-Patterson AFB, Ohio
14 January 1959

To the Editor of Corrosion:

In the December 1958 issue of "Corrosion," there appeared on Page 92 the technical note entitled "Problems of Fuels Containing Boron Discussed by Committee on High Temperature Corrosion." Unfortunately there are several statements in this article which are either erroneous or confusing to the reader.

First, the statement is made that coatings have been developed which extend the life of some metals from minutes to about 150 hours. Not only does this statement fail to mention the high temperatures involved (2000 to 2400 F), but it is in serious error as no such coating is available at this time.

Secondly, the mechanisms of attack reported not only fail to mention all the types of corrosive attack involved, but the metallurgical phenomena described as "eutectic formation" is confusing. Nickel base alloys are susceptible to failure by the formation of nickel boride-nickel eutectics (melting points as low as 1950 F compare to alloy melting temperatures around 2500 F) formed through the diffusion of elemental boron into the alloy lattice. This elemental boron can arise from either of two sources: (1) elemental boron in the B_2O_3 laden combustion products, or (2) from the reduction of molten B_2O_3 by Ti or Al in alloys containing these elements for precipitation hardening purposes. It appears that the Ti and/or Al in the alloy diffuses to the surface, and chemically reduces the molten B_2O_3. The elemental boron thus formed diffuses into the alloy lattice, either interstitially or along the grain boundaries, and combines with the Ni to form the above mentioned low melting eutectics. A similar situation was observed in stainless steels, where the boride phase formed was not Ni, but rather Fe. Other types of corrosion which have been observed include: (1) pitting (2) general surface corrosion, and (3) penetration (either inter or transgranular). No research has been conducted on aluminum or titanium alloys for obvious reasons (2000 F service conditions).

The third confusing statement pertains to the 1500 F testing range reported for the University of Dayton research effort. No significant corrosion of the types reported above occurs at this testing temperature, and corrosion only becomes a problem at temperatures over 1800 F.

It is hoped that this information will be found of some assistance, and if you have any questions please do not hesitate to contact me.

Sincerely,
James R. Myers,
1/Lt., USAF

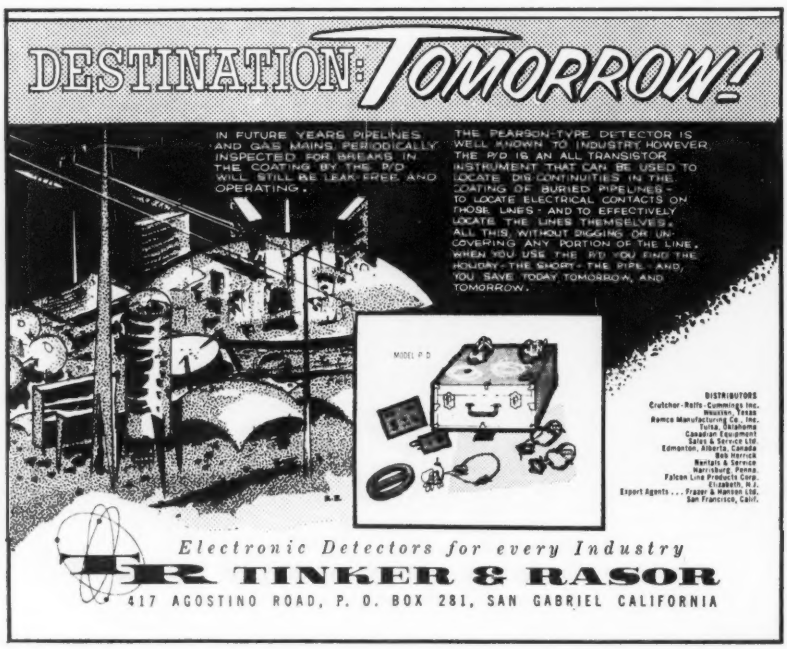
Editor's Note: An extensive discussion of boron oxides was published on page 49 of the February issue of CORROSION.

Rocket Engine Coatings

The following comment by David Roller, Senior Project Engineer, Electrochemical and Corrosion Section, Wright Air Development Center, Wright-Patterson Air Force Base, Ohio, corrects some misstatement in the article "Rocket Engine Coatings May Have Industrial Uses," in December, 1958 CORROSION, Page 92:

Flame sprayed coatings are being developed for possible engine applications. Engines include rocket motors but are not limited to this propulsive device since flame sprayed coatings are also being developed for ramjets, turbojets and afterburners of turbojets as well as a host of other applications. Coatings do not melt "in the ranged (sic) 1500-2000 F." Almost all coating materials being investigated melt over 3600 F (the melting point of alumina) and many melt over about 4500 F.

The method being used experimentally does **not** usually involve "the use of a rapidly moving air stream heated to temperatures in the 1500 F range." A rapidly moving gas stream, either air, an inert gas, or a combustion gas stream, is used to heat the chosen coating material to its melting point and considerably higher. Coating materials, either as a powder or a rod, are deposited as molten particles onto the selected substrate. Usually either a hydrocarbon fueled torch or a constricted electric arc plasma jet torch is used to heat the coating material. Conventional hydrocarbon fueled torches achieve temperatures of the order of 5500 F while arc plasma jet torches develop temperatures above 10,000 F. Particle velocities can vary from about 500 feet per second to about 2000 feet per second for conventional torches while arc plasma torch operation furnishes an intermediate velocity to the molten particles.


DESTINATION: TOMORROW!
IN FUTURE YEARS PIPELINES AND GAS MAINS, PERIODICALLY INSPECTED FOR BREAKS IN THE COATING BY THE P/D WILL STILL BE LEAK-FREE AND OPERATING.
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DEFINITION—
Of a Corrosion Engineer

The definition of a corrosion engineer given below was received from J. D. Ghesquiere, vice president of engineering at the Hinchman Corporation, Detroit 1, Michigan.

The definition came from his engineers during extensive radio noise studies and resulted from an interdepartmental fued between the radio engineers and the corrosion engineers.

Mr. Ghesquiere hopes to find a fitting rebuttal.

"A corrosion engineer is a frustrated individual who passes as an exacting expert on the basis of being able to turn out with prolific fortitude infinite strings of incomprehensible design criteria and formulae calculated with micrometric precision from vague assumptions which are based on debatable figures taken from inconclusive field surveys carried out with instruments of problematical accuracy by persons of doubtful reliability and questionable mentality for the avowed purpose of confounding a hopelessly overworked and underpaid group of commercial wizards referred to, all too frequently, as plant engineering executives."

NACE Abstract Service Adds Two New Sources

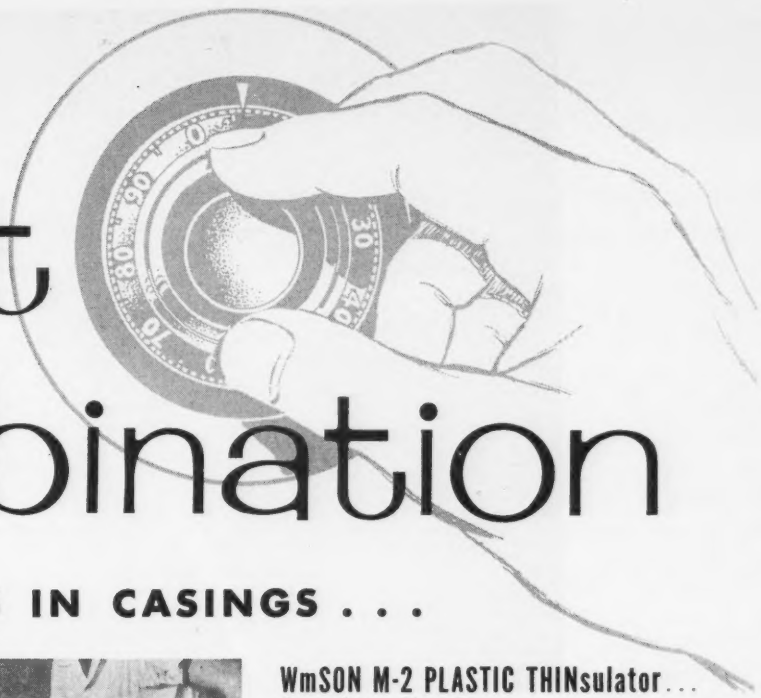
A total of 22 sources are used by the NACE Abstract Punch Card Service to obtain abstracts pertaining to corrosion control and allied fields. Two new sources were recently added: Platinum Metals Review from England and WEAR from Holland.

The 22 sources are from seven foreign countries: Holland, Japan, Mexico, Canada, England, Germany and India. Russian material is abstracted through a translation agency in this country.

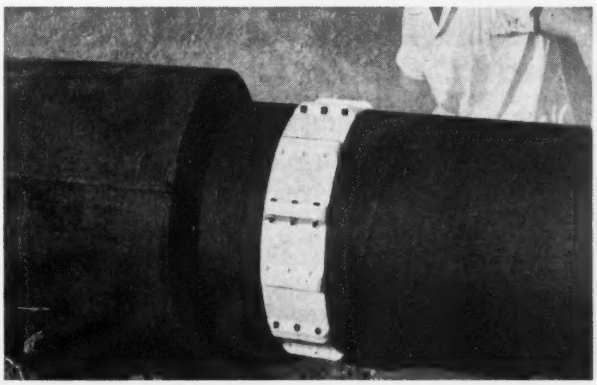
Demonstration of the use of abstract punch cards will be given during the Chicago Conference at the NACE booth.

Over 20,000 abstract cards have been issued by the Abstract Punch Card Service.

the right combination



FOR PIPELINES IN CASINGS . . .



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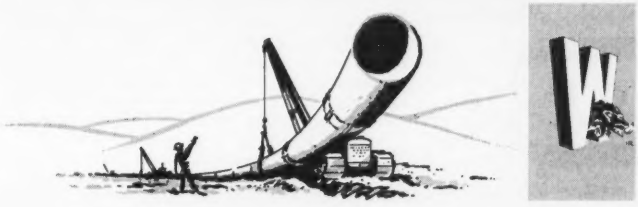

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Mild fumes, splash or spillage of most acids and alkalies. Continuous temperatures to 150°F. where maximum surface preparation is not practical. Structural steel, pipe, walls, equipment exteriors. Carboline CS-200 (all vinyl) system.

CONDITION 2

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CONDITION 3

Heavy duty protection against solvent, caustic and acid spillage. Continuous temperatures to 200°F. Structural steel, equipment exteriors, concrete. Carbomastic #3 (epoxy tar) and Phenoline 305 topcoats.

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Write for newly released Chart No. 5 showing comparative ratings of coating systems by physical and chemical properties — an engineering aid in choosing the most efficient protection. Also ask for technical data and recommendations on Carboline maintenance systems.

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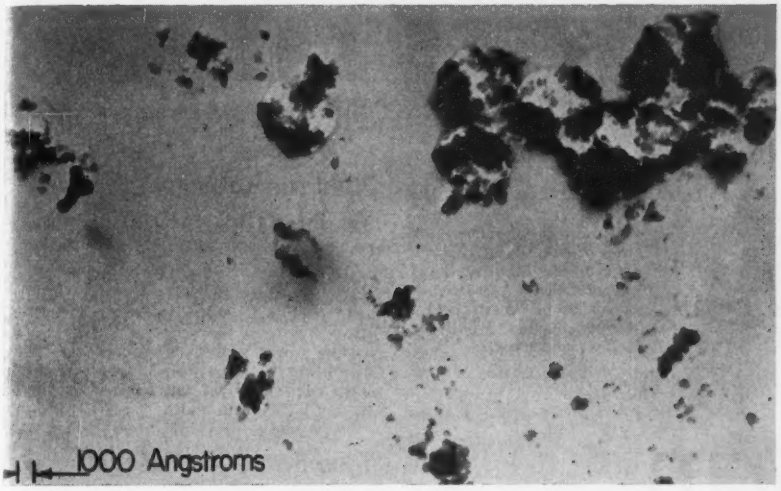
GENERAL NEWS



ULTRA-FINE METAL POWDERS are being produced by a newly discovered method. At a magnification of 50,000 diameters, individual aluminum particles are shown above to be spheroidal in shape with an average diameter of about one-millionth of an inch. Larger dark areas are piles of tiny particles which were not spread when the sample was prepared for examination. Individual particles are scattered and appear as small dark spots.

Ultra-Fine Powders Are Produced Using New Process

A newly discovered method for producing ultra-fine metal powders with particles one-millionth of an inch in diameter has been announced by the National Research Corporation, 70 Memorial Drive, Cambridge 42, Mass.

The new powders may make possible unknown processes and applications in the catalytic chemical process, powder metallurgy and other industries. Chemical significance is the potential ability to enter catalyzing chemical reactions in which the same metals in coarser states will not do unless subjected to high pressures and temperatures or both.

In powder metallurgy, these powders may offer short cuts to new exact-composition alloys through the sintering process with better physical properties than in present alloys, according to the NRC.

Features and properties of aluminum fine metal powder have been given by NRC:

1. Increased surface energy and surface area: The figure shows particles to be 0.005 to 0.06 microns with an average diameter of 0.03 microns (approximately one-millionth of an inch). Smallest aluminum particles presently available are 6.0 to 10.0 micron in size.

2. Surface area is about 750,000 square centimeters per gram.

3. In some catalytic chemical processes, lower reaction thresholds (temperature and pressure), high reaction rates and increased yields may be attainable with resulting decreased equipment costs.

4. Extreme reactivity, large surface area and thixotropic nature offer possible uses in high energy chemical reactions.

5. Dry bulk density is 0.08 grams per cubic centimeter (5 pounds per cubic foot) and tamped density is 0.22 grams (14 pounds per cubic foot).

6. Particles are spheroidal in shape.

NACE Members to Give Papers at AIChE Meeting

Four NACE members will present two papers of probable interest to corrosion engineers during the March 15-18 39th National Meeting of the American Institute of Chemical Engineers to be held in Atlantic City, N. J.

The papers are "Use of Plastics for Corrosion Protection in the Chemical Industry" by H. W. Schmidt and A. R. Gabel, both of Dow Chemical Co., Midland, Mich., and "Use of a Modified Flexure Tester to Estimate Strength Changes in Plastics Due to Corrosive Exposures" by R. P. Lee and D. L. Hawke, both of National Lead Co., South Amboy, N. J.

Air Pollution Research

Two more years of intensive scientific work can provide the essential facts needed for final solution of the Los Angeles smog problem, according to W. L. Faith, managing director of the Air Pollution Foundation.

Private industry can use the foundation's data to produce workable controls to eliminate smog, he added.

Armour Foundation to Hold Inhibitor Symposium May 7

A Symposium on Corrosion Inhibitors will be held in Chicago, sponsored by the Armour Research Foundation of Illinois Institute of Technology and the Chicago section of the Electrochemical Society.

Co-chairmen for the symposium are Ernest L. Koehler, NACE member associated with Continental Can Co., and Jacob Miller, Armour Research Foundation.

Basic studies of the theory and mechanism of inhibitory processes will be considered.

Additional information can be obtained from M. J. Jans, Armour Research Foundation, 10 W. 35th Street, Chicago 16.

ASEE and AEC to Sponsor Nuclear Energy Institute

Six Summer Institutes on Nuclear Energy for engineering educators are scheduled under the sponsorship of the Atomic Energy Commission and the American Society for Engineering Education.

Purpose of the institutes is to provide special training in the fields of nuclear energy and the nature of nuclear reactor problems.

Basic institutes will be held at North Carolina State College and Cornell University; advanced institutes at University of Michigan, Argonne National Laboratory, Hanford Laboratories and Pennsylvania State University.

Appointment applications can be obtained from deans of engineering at universities or from ASEE headquarters at the University of Illinois, Urbana.

ASEE Helps India Plan New Engineering School

Six American representatives from engineering teaching have been named by the American Society for Engineering Education to study plans for a new engineering school at Kanpur, India.

The study was undertaken at the request of the Indian Ministry of Scientific Research and Cultural Affairs and is being conducted under a contract with the U. S. International Cooperation Administration.

Houston Paint Convention

Two days of technical meetings are scheduled for the April 23-25 Southwestern Paint Convention to be held at the Shamrock Hilton Hotel, Houston.

Convention theme is "New Products, New Markets—Through Creative Technology." A Raw Material and Equipment Show is also planned.

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Exhibits at the 1959 Corrosion Show will be staffed by experienced corrosion engineers qualified to answer questions on corrosion control.

Air Pollution Report

Auto exhaust elements that must be eliminated and the fact that changes in gasoline will not reduce smog are discussed in the Air Pollution Foundation's fifth annual report.

Also included is a discussion of the work being done on exhaust control devices.

Electrochemical Society

A meeting of The Electrochemical Society, Inc., is scheduled for May 3-7, 1959 in Philadelphia. Sessions are planned on electric insulation, electronics, electrothermics and metallurgy, industrial electrolytics and theoretical electrochemistry.

AEC Patents Released

Descriptions of 60 patents owned by the U. S. government and held by the AEC have been released. Included are the following: 2,843,478 on high temperature brazing alloy for joining Fe-Cr-Al materials and austenitic stainless steels; 2,843,500 on coated alloys; 2,846,762 on metal plating process, 2,847,321 on metal surface treatment and 2,848,351 on nitric acid pickling process.

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PERIODICALS

Pinturas y Acabados Industriales. (In Spanish) First Issue, January, 1959. Published by Pinturas y Acabados Industriales, Muntaner 171, Barcelona, Spain. Annual subscription (6 issues) 100 pesatas.

To contain short and practical articles on organic and metallic coatings and news on paint technology and galvanizing.

Nuclear Power Association To Test-Operate Reactor

Carolinas Virginia Nuclear Power Associates will test operate a heavy water, moderated pressure tube-type reactor fueled with slightly enriched uranium.

The plant will produce steam for an adjoining conventional power station. The net electrical capacity is expected to be 17,000 kilowatts.

Two Societies Merge

Two engineering societies have voted to merge. The American Society of Heating and Air-Conditioning Engineers and the American Society of Refrigerating Engineers have joined as ASHRAE, American Society of Heating, Refrigerating and Air-Conditioning Engineers.

Reactor Becomes Critical

Spert-III (Special Power Excursion Reactor Text No. 3), a versatile research facility developed for studying nuclear reactor safety and operated by Phillips Petroleum Company for AEC, recently achieved criticality at the Idaho Falls Station.

Welding Information

An Information Center has been established by the American Welding Society, 33 West 39th St., New York City, to serve as an authoritative source for information related to welding.

ASTM Book of Standards

The first of 10 parts of the ASTM's 1958 Book of Standards has been published. It was on non-ferrous metals. Other parts of the book are scheduled for publication later.

Conference on Titanium

A two-day conference on Titanium Metallurgy has been scheduled September 14 and 15 by New York University's College of Engineering.

X-Ray Analysis Conference

The 8th Annual Conference on Applications of X-Ray Analysis sponsored by the Metallurgy Division of the University of Denver's Research Institute will be held at Estes Park, Colo., August 12-14. More information can be obtained from William M. Mueller at the University of Denver.

Corrosion control unlimited! No other supplier offers as many materials or as much practical experience in corrosion control. Federated's Corrosion Advisory Service can recommend the best for you from among magnesium and zinc; GALVANIC ANODES, for cathodic protection; LEAD SHEET, PIPE and FITTINGS; ZINC and ZINC ALLOYS; COPPER and ALLOYS; and PLATING MATERIALS, including copper, lead, cadmium, zinc and silver anodes; nickel salts and addition agents for plating baths. One of Federated's 22 sales offices is near you. Don't hesitate to call for advice. Federated Metals Division, 120 Broadway, New York 5. In Canada: Federated Metals Canada, Ltd., Toronto and Montreal.

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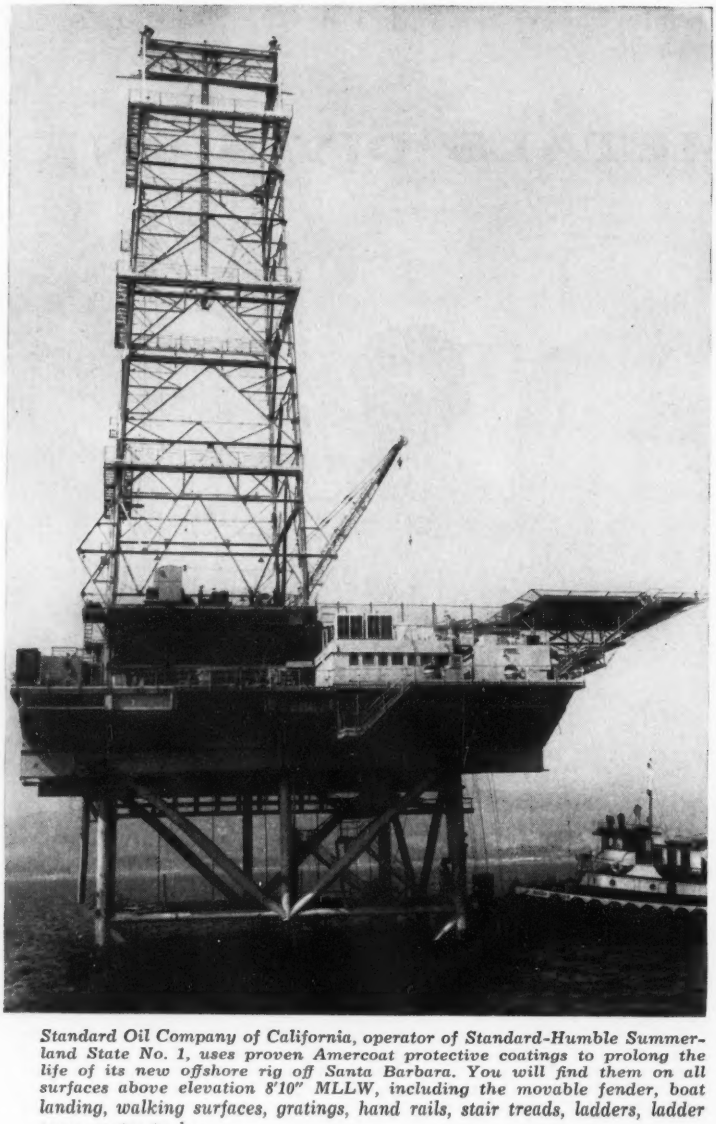
This offshore rig stands in the

WORLD'S LARGEST ELECTROLYTE



Standard Oil Company of California, operator of Standard-Humble Summerland State No. 1, uses proven Amercoat protective coatings to prolong the life of its new offshore rig off Santa Barbara. You will find them on all surfaces above elevation 8'10" MLLW, including the movable fender, boat landing, walking surfaces, gratings, hand rails, stair treads, ladders, ladder cages, water tanks.

The Pacific is terrific—for boaters and bathers. But plant an unprotected steel structure in it, and the world's largest electrolyte begins its attack. In a short time, spray, mist and wave action reduce steel surfaces to the status of a spent battery plate.

Such will not be the fate of California's first permanent offshore drilling rig because modern protective coatings completely shield all steel above the water line from moisture and sea atmosphere.

A permanent base coat of Dimetcote, the inorganic zinc silicate coating, armors all vulnerable surfaces with a tough zinc film that bonds chemically and physically to the steel. If minor abrasions occur, the zinc also doubles as a sacrificial anode to prevent corrosion of the exposed metal. As further defense against the sea, the Dimetcote is covered with Amercoat protective vinyl coatings which add chemical resistance and film thickness.

Will this Amercoat protective coating system stand up against the ravages of the Pacific? It should, for it has performed successfully on scores of offshore rigs, barges and related equipment operating in the Gulf of Mexico and other salt water bodies.

Next time you have a project involving seagoing steel, why not discuss protective coatings with us? Our decades of experience and thousands of case histories may save you time and money in your planning.

For detailed information, technical data and assistance with your marine corrosion problems, write to any of the Amercoat offices listed below:

115

- 921 Pitner Avenue, Evanston, Illinois
- 2404 Dennis Street, Jacksonville, Florida
- 360 Carnegie Avenue, Kenilworth, New Jersey
- 6330 Supply Row, Houston, Texas

Amercoat® CORPORATION

Dept. GC1, 4809 Firestone Blvd.
SOUTH GATE, CALIFORNIA

Equipment Services NEW PRODUCTS Materials Literature

Aluminum

Aluminum Transformers developed jointly by Moloney Electric Co., St. Louis, and Aluminum Company of America, Pittsburgh, Pa., have been shown at the American Institute of Electrical Engineers. The transformer, made of aluminum except for the core and insulation, is the first distribution transformer to be shown that uses aluminum strip conductors. The coil is wound with strips of aluminum foil, substituted for the usual copper wire.

•

Thick-jointed Aluminum Pipe developed by Reynolds Metals Co., Richmond 18, Va., is being used by the Superior Oil Company in Venezuela. The pipe is extruded to provide extra thickness at pipe ends.

•

Aluminum Magnet Wire in a range of sizes, shapes and insulations is being produced by Kaiser Aluminum & Chemicals Sales, Inc., 919 North Michigan Ave., Chicago 11. The wire is reported to have improved winding characteristics and better performance at high temperatures than copper.

•

Booklets describing the mill products of Kaiser Aluminum, 919 North Michigan Ave., Chicago 11, are available from that company. Included are various types of sheet, plate, foil, rod, bar, wire, extrusions, tubing, forgings, billet and pig.

•

Aluminum and Magnesium Forging facilities of Alcoa are described in a new booklet available from Aluminum Company of America, Pittsburgh, Pa.

Alloys

Iron-Aluminum-Manganese alloys combining light weight, cold workability, high temperature strength and oxidation resistance have been developed by the National Research Corp., 70 Memorial Drive, Cambridge 42, Mass. The research was sponsored by the Navy's Bureau of Aeronautics to consider the alloys for possible high temperature uses in missiles, aircraft and automotive industries.

•

Engineering Properties of Ni-Resistant ductile irons are given in a booklet available from Reader Service Dept., International Nickel Co., Inc., 67 Wall St., New York 5. It describes the metal's strength and resistance to heat, wear and corrosion.

•

Heat Resistant Alloys three to four times as strong as conventional metals at high temperatures have been developed at the Denver Research Institute, University of Denver. The new process used involves mixing ceramic materials with metals. Copper has been produced that is three or four times as strong as conventional copper at 1800 F, and aluminum at least three times as strong as conventional aluminum at 1000 F, according to the Institute.

Five Wear-Resistant Alloys in the form of investment castings are described in a booklet available from Haynes Stellite Co., Division of Union Carbide Corp., Kokomo, Ind. Chemical, physical and mechanical properties of the four cobalt-base and one iron-base alloys are given.

Cathodic Protection

Trailing Platinum-Clad tantalum nickel anodes used to protect the hull of S. S. Israel, a 10,000-ton passenger-cargo ship between Haifa and New York will be marketed in the U. S. by Metals and Controls Corp., Attleboro, Mass. The anodes (described in a CORROSION article in February, 1957) and the method used to control the immersed portion of ship hulls is patented by Herman S. Preiser, corrosion consultant to the U. S. Navy Bureau of Ships. The anode, trailing behind and beneath the ship, is connected to the positive terminal of a welding generator whose negative terminal is grounded to the ship's hull. Current required to polarize the hull on the S. S. Israel was 50 amperes at 20 volts.

Coatings

Fire-Retardant Coatings for thermal insulation materials that are also corrosion resistant are described in a folder available from Royston Laboratories, Inc., Pittsburgh 38, Pa.

•

Elastomer 614, a pre-polymerized one-part allophonate protective coating, has been introduced by Howley Chemical Co., Inc., 26 Howley St., Peabody, Mass. Available in clear, colorless and color pigmented forms, the coating is reported to have above average film strength, unusual resistance to chemical attack, high gloss and good adhesion to a variety of surfaces.

•

Cadmium Coating Process, developed by NRC Equipment Corp., 160 Charlemont St., Newton, Mass., protects high tensile steels against corrosion without danger of hydrogen embrittlement. Samples passed 96-hour salt spray and salt bath tests without traces of corrosion, according to NRC reports.

•

Transocean will be the trademark for products manufactured by the 10 European companies joined in the Transocean Marine Paint Association. The adopted charter proposes a free pooling of technical research and laboratory control of a full range of products under one trademark and marketing policy.

•

New Hot Spray Vinyl that gives five mils of protection in one spray coat has been announced by Gates Engineering Co., Wilmington 99, Del. It is a high solids vinyl solution formulated for hot spray applications that are quick drying with little overspray, good edge built-up and resistant to sagging.

•

Wet Ground Mica increases moisture resistance and improves the corrosion protective characteristics of vinyl alkyd paints according to a study given in Technical Bulletin No. 37 available from the Wet Ground Mica Association, Inc., 420 Lexington Ave., New York 17.

•

Variable Ratio Resin Dispensers for use with most two-component liquid resin and hardener systems has been announced by the Delsen Corp., 719 West Broadway, Glendale 4, Cal. It permits rapid change from one resin-hardener system to another and changes in resin to hardener ratio to compensate for ambient temperature changes.

•

Alcoa Stabilizer No. 5, a new additive that eliminates deleafing of aluminum pigments in alkyd and high-acid varnish vehicles, has been perfected by Alcoa Research Laboratories, New Kensington, Pa. The additive is reported also to increase paint durability and corrosion resistance to salt water and salt water spray.

•

Alkydol S-1718, a polyester type coating resin, is described and formula suggestions and evaluation of enamels given in a bulletin available from Alkydol Laboratories, Inc., 3242 South 50th Ave., Cicero 50, Ill.

•

Coal Tar Protective Coatings produced by Pittsburgh Coke & Chemical Company will be distributed in the Louisville area by General Insulation & Roofing Co., Inc., 116 North 3rd St., Louisville 2, Ky.

•

Protective Coatings Bulletin, available from Carboline Company, 32 Hanley Industrial Court, St. Louis 17, Mo., gives a breakdown of four classifications of corrosive conditions according to severity of exposure, service requirements, physical properties and typical recommendations.

•

Napko Protectives are described in a new booklet available from Napko Corp., P. O. Box 14126, Houston 21, Tex. Classification, gloss, application, thinning ratios, drying time, coverage and color reference are given.

Consolidations

LOF Glass Fibers Company has been purchased by Johns-Manville Corporation and will be called Johns-Manville Fiber Glass Inc., with plants at Parkersburg, W. Va., Houston, Tex., Corona, Cal., and Waterville and Defiance, Ohio.

•

Combustion Engineering, Inc., has acquired General Nuclear Engineering Corporation and will operate it as a subsidiary.

•

Rome Cable Corporation and Aluminum Company of America have announced plans to affiliate. They plan to combine their electrical conductor, conduit and accessory activities in a new company to be called Rome Cable Company.

Instruments

Rio Soil Selector, a new instrument to simplify 4-pin soil resistivity measurements for cathodic protection work, is being produced by Rio Engineering Co.,

(Continued on Page 106)

NEW PRODUCTS

(Continued From Page 105)

P. O. Box 6035, Houston 6, Tex. Various measurements can be made by rotating a six-position selector switch. Vulcanized, multi-conductor cable with properly spaced take-outs eliminates tape measurement for proper pin spacing.

•

New Immersion Heaters made of steel, stainless steel and translucent fused quartz with their own thermostats are being produced by N. J. Thermex Co., Inc., 535 Bergen St., Harrison, N. J. They are designed for small plating tanks where only one heater and thermostat are required.

•

Cold Cathode Principle has been used in a new high-vacuum gage for pressure measurement developed by Miller Laboratories, P. O. Box 97, Brea, Cal. Operated by ionization of gas molecules present in a vacuum system, the gage can be used in hermetic sealing, vacuum pump-down systems, metal evaporation, mass spectrometry, vacuum distillation, vacuum metal refining and as a general instrument for low pressure measurements.

•

Model M-1 Holiday Detector has been re-designed by Tinker & Rasor, P. O. Box 281, San Gabriel, Cal. It will be housed in two plastic covered cases. The signal bell and relay have been enclosed, and the bell battery has been changed to standard flashlight batteries.

A Soil Moisture Meter designed to indicate the amount of moisture available in a given section of soil, is described in a bulletin available from Soiltest, Inc., 4711 W. North Ave., Chicago 39.

Laboratories

Koppers Company, Inc., has begun construction of a new Research Center at Monroeville, Pa. It will include an administration building, three chemical labs, power plant and supporting facilities.

•

Carpenter Steel Co., Reading, Pa., is now operating a new metallurgical control laboratory to provide faster service in production testing of steels to customer specifications.

•

Du Pont's Electrochemicals Department has opened a new research center for its work in sodium, vinyl products, peroxygen compounds, nylon intermediates and related fields. The center is in Niagara Falls, N. Y.

•

Flexonics Research Laboratories, a new division of Flexonics Corp., have been established at Elgin, Ill. It will provide testing and development service on a contract basis to industry and to development agencies of the government.

Metals—Ferrous

Type 304L Stainless Steel containing 2 percent boron melted in electric arc furnaces is now available commercially from Carpenter Steel Co., Reading, Pa. This steel is used in the commercial atomic energy field for control rods and as a shielding material.

4-D Wrought Iron for building drainage systems is discussed in a new booklet available from A. M. Byers Co., P. O. Box 1076, Pittsburgh 30, Pa. It describes corrosive conditions encountered in drainage services, service records, typical installation and performance tables.

•

Wire Cloth Problems are discussed in a booklet available from International Nickel Co., Inc., 67 Wall St., New York 5, N. Y. Physical advantage of wire cloth, range of weaves and sizes available and mechanical properties are given.

•

Cast Iron Pressure Pipe of 6 to 16-inch diameter in 18-foot lengths are being manufactured by the metal mold process at the new Chicago plant of James B. Clow & Sons, Inc.

Plastics

Colonial Supply Co., Pittsburgh, Pa., has been appointed sales distributor of A. M. Byers Company's PVC plastic pipe. The company also distributes Byers 4-D wrought iron pipe.

•

Solid Polyurethane Applications are described in a booklet available from Disogrin Industries, Inc., 510 South Fulton Ave., Mount Vernon, N. Y. Based on a paper presented at the 1958 ASME annual meeting, the booklet reviews the fabrication of parts from solid polyurethane and discusses industrial applications.

•

PVC Piping, Fittings and Valves made of rigid Koroseal polyvinyl chloride are

(Continued on Page 108)

NEW • COMPACT
LIGHTWEIGHT • PORTABLE
INSTANTANEOUS READINGS
SINGLE KNOB CONTROL

LABLINE, Inc.,
3070-80 W. Grand Ave. • Chicago 22, Ill.

If you have corrosion problems then you need
LABLINE-PURE
CORROSION TESTER
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For FIELD or LABORATORY

Single Knob Control gives direct readings. Detects corrosion of less than .0000002 inches. No shutdowns necessary. No need for separate temperature corrections. Rugged, shock-proof, all-transistor circuit. Battery operated. All types of probes available with wide range of uses. Write for full information.

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Probes Extra

Developed by The Pure Oil Company Research Center
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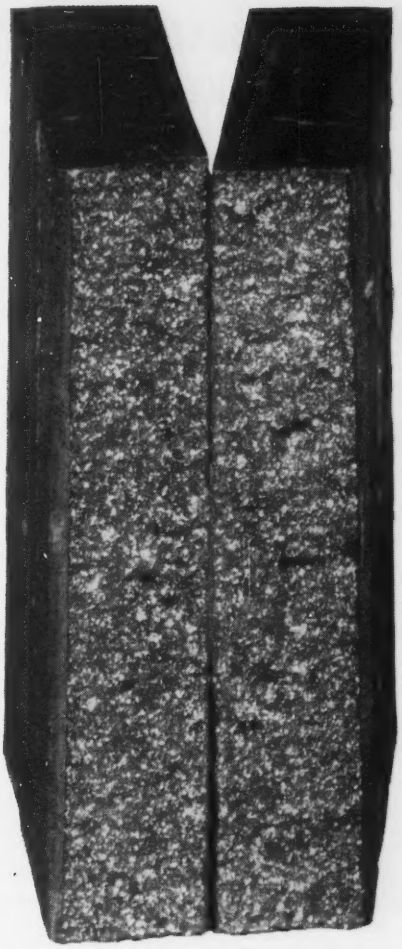


4-D Wrought Iron **Steel**

HERE IS THE DIFFERENCE

These are actual photographs of fractured pieces of 4-D Wrought Iron and steel. Photo at left shows how the fibrous structure of 4-D Wrought Iron differs from that of steel.

Many thousands of glasslike iron silicate fibers entrained in the pure base metal account for 4-D Wrought Iron's unique structure. Duplicated in no other metal, this structural feature gives 4-D Wrought Iron its superior resistance to corrosion and fatigue. Superior, even, to standard wrought iron.

Increased deoxidation of the base metal, greater phosphorous content, and the use of more siliceous iron silicate account for new 4-D Wrought Iron's acceptance as the most versatile material yet developed to help you fight salt water corrosion.

While initial cost may be higher, case histories and recent laboratory tests prove 4-D Wrought Iron the most economical buy because of its real cost: cost per year of service.

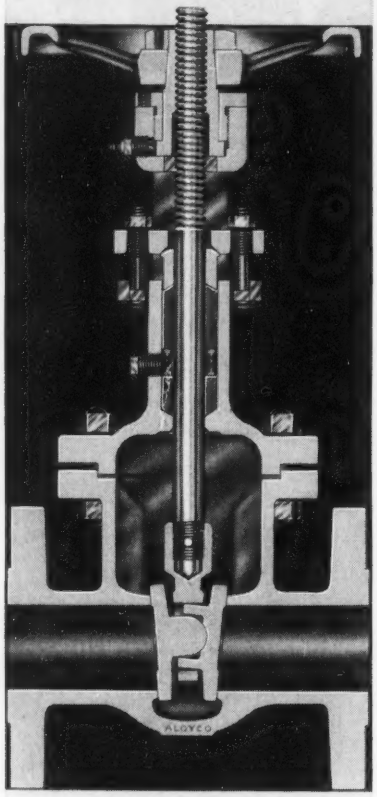
Write us for comparative test results and new 4-D Wrought Iron literature. Let us prove to you how conclusively this metal outperforms ferrous substitutes in a wide range of marine applications. A. M. Byers Company, Clark Building, Pittsburgh 22, Pennsylvania.

BYERS 4-D WROUGHT IRON

TUBULAR AND FLAT ROLLED PRODUCTS

ALSO AMBALLOY ELECTRIC FURNACE STEELS AND PVC PIPE AND SHEET

FOR HIGH-PRESSURE CORROSIVE SERVICE:



300 lb. Aloyco Stainless Steel Gate Valves

Figure 2117 double disc. (above) Available in sizes ½" to 8".
Figure 2217 for solid wedge. Sizes ½" to 12".
In addition—screwed, and socket weld ends. Sizes ½" to 2".
Other 300 lb. designs including globes, swing and lift checks; jacketed and tank valves.
600 lb. designs also available.

Materials
Types 304 and 304L
Types 316 and 316L
Aloyco 20
Monel and nickel
Hastelloy alloys B and C

For more information on Aloyco valves for your specific corrosive service, write for Bulletin #7 to Alloy Steel Products Company, 1301 West Elizabeth Ave., Linden, New Jersey...the one manufacturer specializing in Stainless Steel Valves exclusively. 9.3

Longer Lasting ALOYCO VALVES
FIT IN CORROSIVE SERVICE

ALLOY STEEL PRODUCTS COMPANY
1301 West Elizabeth Avenue
Linden, New Jersey

NEW PRODUCTS

(Continued From Page 106)

described in a new catalog available from Plastic Products Division, B. F. Goodrich Industrial Products Co., Marietta, Ohio. It lists specifications and physical properties of Koroseal piping and gives data on chemicals that can be handled by the piping.

Pumps

Corrosion Resistant Liquid Ends are used on the Series 100 controlled capacity pumps developed by American Meter Company's Pump Division, 13500 Philmont Ave., Philadelphia 16. The ends are available in carbon and high alloy steels.

•

Continental Utility Pumps using the helical screw principle without vanes, turbines or impellers are described in a catalog available from Continental Pump Co., 1027 South Vandeventer, St. Louis 10, Mo.

Solar Energy

The Solar Research House in Tucson, built by the Institute of Atmospheric Physics of the University of Arizona and the Copper and Brass Research Association, will be heated and cooled by a solar energy system. The system is designed for houses in the $16,000 to $18,000 price range and may be capable of heating and cooling a home for half the cost of conventional systems.

Solvents

Non-Flammable Trichlorethylene is being used as a vapor degreaser and paint thinner by Du Pont Electrochemicals Department, Wilmington, Del. It permits safer, faster and more economical techniques for cleaning and painting metal parts, according to the company.

Surface Preparation

Clementina, Ltd., traveling sandblasting school opens a tour of the southern states in March. Times and locations can be obtained by writing the company at 2277 Jerrold Ave., San Francisco 24, Cal.

Tape

Seamless Rubber Co., New Haven, Conn., has announced a new system for application of polyethylene tape as a pipe coating. A prime coat is applied to the pipe prior to application of the pressure sensitive polyethylene tape.

•

Polyken Tape will be used on the 1616-mile pipeline running from Baton Rouge, La., to Miami, Fla. This is believed to be the first major pipeline to be completely wrapped with tape. Polyken is produced by Kendall Company, 309 W. Jackson Blvd., Chicago 6.

Tubing

Tube Manufacturing skills and techniques are described in a new brochure available from Wolverine Tube, 17200 Southfield Road, Allen Park, Mich. It discussed the manufacture of seamless copper and copper alloy tubing.

•

Stainless Steel Tubing Catalog has been revised by Tube Turns, Louisville 1, Ky. It gives dimensional data on welding fittings and flanges, their allowable stress values, corrosion resistance and pressure-temperature ratings.

Valves

Hastelloy Valves in Alloys B, C and D are described in Bulletin No. 10 available from Alloy Steel Products Company, Inc., Linden, N. J.

Welding

New Wall Chart showing the most suitable welding rod for a given job has been designed for workmen and supervisors by All-State Welding Alloys Co., Inc., White Plains, N. Y.

•

High Speed Hand Welders for thermoplastics are described in a new brochure available from Laramy Products Co., 90 South St., Hingham, Mass. Information on welding speeds and strengths, characteristics of construction and lists of accessories are included.

MEN in the NEWS

Hack Arroe has been appointed as head of the Physics Division of the Denver Research Institute of the University of Denver.

•

Wiser Brown, chief industrial engineer for Aluminum Company of America, has retired after 36 years with Alcoa.

•

John J. Dailey, group engineer with Temco Aircraft Corp., Dallas, was awarded $5000 for his development of a new process to remove oxide scale from titanium metal parts.

•

Robert H. Ebersole has been named district manager of the Detroit sales office of Federated Metals Division of American Smelting and Refining Company.

•

F. G. Fabian, Jr., has been promoted to president of Dresser Manufacturing Division, Bradford, Pa.

•

Howard L. Gerhart has been appointed director of research and development for the Paint and Brush Division of Pittsburgh Plate Glass Company.

•

F. C. Langenberg and **G. E. Hutchinson** have been appointed material and process engineers by the Crucible Steel Company of America, Pittsburgh, Pa.

•

John H. Ingalls is a new field service engineer at the A. M. Byers Company's Boston Division.

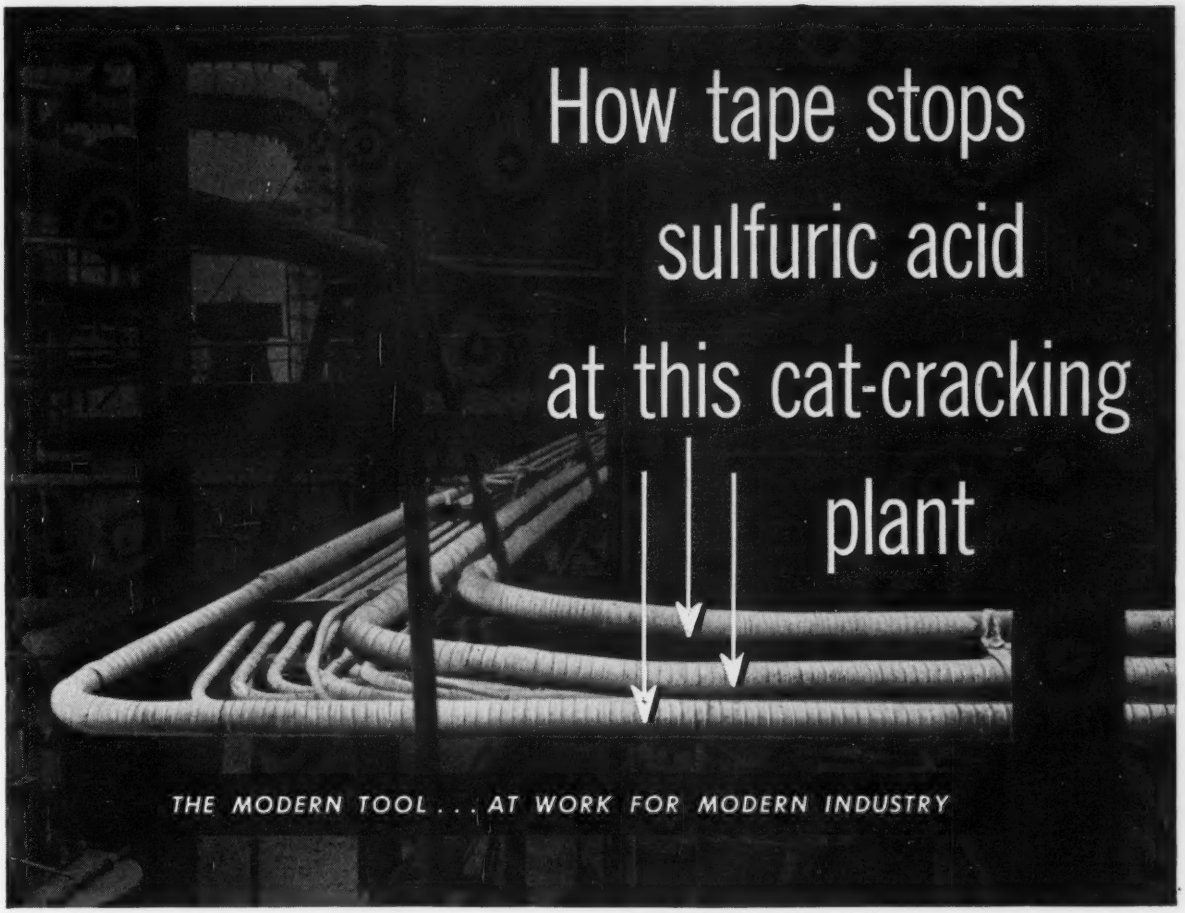
•

Mrs. Laurence Pellier, research metallurgist, has joined the staff of the Research Laboratory at International Nickel Co., Bayonne, N. J.

•

Alan H. Brown has been assigned to Eastern Venezuela as field engineer with South American Petrolite Corporation.

(Continued on Page 110)



FIVE YEARS UNDER THESE HIGHLY CORROSIVE CONDITIONS PROVES THAT POLYKEN PROTECTION WORKS . . . *TO TRIM COSTS*

Look in on one of the world's largest fluid catalytic cracking units. A maze of conduit and pipe exposed to an extremely corrosive sulfuric acid atmosphere.

Formerly, these structures had to be painted every three or four months. Maintenance costs were excessive. Then, five years ago, they were wrapped with Polyken Protective Tape Coating. They have needed practically no attention since.

INERT POLYETHYLENE

The reason is that Polyken takes amazingly inert polyethylene and makes it into a tape coating with all these properties.

- **tough and elastic**
- **unplasticized, non-drying film**
- **doubly thick adhesive to seal all voids**
- **far higher adhesion for a true bond to pipe surface**
- **lower water vapor transmission rate**
- **higher electrical insulation resistance**
- **better cold weather handling and durability**

Check the savings. Contact the Polyken distributor in your area.

Atlanta, Georgia: Steele & Associates, Inc.
Chicago, Ill.: Sales Engineering, Inc.
Cincinnati, Ohio: Hare Equipment
Cleveland, Ohio: The Harco Corp.
Denver, Colo.: Patterson Supply Co.
Des Moines, Ia.: The Donald Corp.
Fort Worth, Texas: Plastic Engineering & Sales Corp.
Gretna, La.: Allen Cathodic Protection Co.
Houston, Texas: Cathodic Protection Service
Kansas City, Mo.: Industrial Coating's Engineering Co.
Long Beach, Calif.: Barnes & Delaney
Minneapolis, Minn.: Simcoe Equipment Co.
Philadelphia, Pa.: Harold N. Davis Co.
Pittsburgh, Pa.: Royston Laboratories, Inc.
Plainfield, New Jersey: Stuart Steel Protection Corp.
St. Louis, Mo.: Shutt Process Equipment Corp.
San Francisco, Calif.: Aetna Sales Co.
Seattle, Wash.: Farwest Corrosion Control Corp.
Seattle, Wash.: Pacific Water Works Supply Co.
Tulsa, Okla.: William Cluff Corp.

Polyken®
Experienced in modern PROTECTIVE COATING
THE KENDALL COMPANY
Polyken Sales Division

Chemicals Causing Floor Failures?

Here's An Invitation to Consider . . .

TRUSCON CHEMFAST CONCRETE

CHEMICAL RESISTANT FLOORING

When designing or replacing floors for CHEMICALLY CRITICAL AREAS!

- ✻ High Impact Resistance and Load Bearing Strength
- ✻ No Water in Mix Design
- ✻ Integral Resin Binder Protects Against Chemical Attack
- ✻ Positive Bond—No Mortar Joints
- ✻ Saves Repeated Shutdowns
- ✻ Saves Structural Floor Base
- ✻ Placed Like Regular Concrete (Not a Skim Coat)

See Chemfast Concrete in action at Booth 71, Corrosion Show, March 16-20

A request on your letterhead stating your floor corrosion problem will be followed by a personal inspection and specification by a qualified Truscon representative.

TRUSCON Laboratories

Industrial Maintenance Division of Devoe & Raynolds Co., Inc.

Detroit 11, Michigan

MEN in the NEWS

(Continued From Page 108)

Philip E. Sharr has been appointed barium project manager at Columbia-Southern Chemical's plant in South Charles, W. Va.

•

Charles W. Scamman, NACE member, is now associated with the sales division of Cathodic Protection Service.

•

Hillard H. Presley is a new industrial coatings engineer at Napko Corp., Houston.

•

G. S. Martin, NACE member formerly associated with Container Laboratories, Inc., is packaging research coordinator with Douglas Aircraft Co., Inc., Santa Monica, Cal.

•

F. L. Moffet, and **A. P. Terrile** have been appointed to new positions in the metallurgical division of Crucible Steel Company.

•

David H. Jones, NAME member, has joined Cathodic Protection Service's staff and will be in charge of the company's new Chicago office.

•

James L. Henderson will be sales engineer for J. M. Tull Metal & Supply Co., Atlanta, to handle Fansteel metals and fabrications.

•

Edgar H. Dix, Jr., NACE member and retired assistant director of Alcoa research, has received the Navy's Distinguished Public Service Award.

•

T. E. Detcher, NACE member, has been appointed sales manager for the John W. Rouse Construction Corp., Gouverneur, N. Y.

•

Lawrence W. Cooper is the new director of metallurgy at Jessop Steel Co., Washington, Pa.

•

Robert L. Adair, NACE member, has been appointed supply division sales manager for the Houston office of Corrosion Rectifying Company.

•

Bethel Bond, NACE member, is president of the newly organized Rem Corp., 809 West Davis, Conroe, Texas, which will be in cathodic protection engineering and design work.

•

Schrade F. Radtke will direct the joint research program of the American Zinc Institute and the Lead Industries Association.

•

Virgil H. Disney, director of electrical engineering at Armour Research Foundation, was elected president of the 1959 National Electronics Conference to be held October 12-14 in Chicago.

•

James F. Connelly, Sr., will be general manager of A. O. Smith Corporation's new reinforced plastics division in Milwaukee.

•

Over 70 technical committees will meet during the 15th Annual NACE Conference in Chicago.

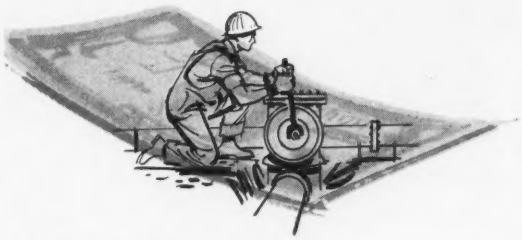
•

Exhibits at the 1959 Corrosion Show will be staffed by experienced corrosion engineers qualified to answer questions on corrosion control.

Save dollars 4 ways with UNICOR®

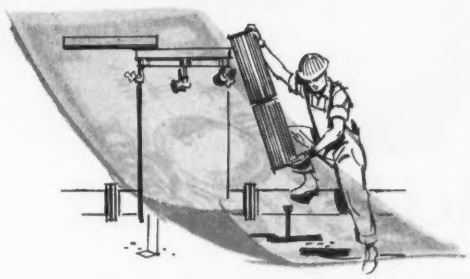
1. Maintain flow capacity

Unicor prevents corrosion in pipelines—keeps pipelines free of scale. With Unicor you are assured of maintaining maximum flow capacity, C factor and output.



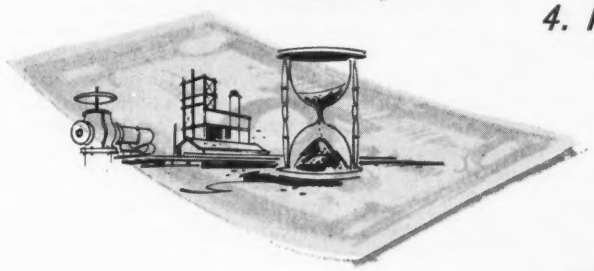
2. Reduce downtime

Downtime to scrape lines and replace filters is production time lost. Prevent such loss with Unicor. It keeps equipment free of products of corrosion.



3. Cut filter replacements

What's your annual bill to service and replace filters? A pretty penny, no doubt. You can drastically reduce this expense by using Unicor.



4. Increase equipment life

Tanks, vessels and pipelines stand up longer when you give them effective protection against corrosion. A little Unicor goes a long way—protects against corrosion from well to consumer.

Unicor, the preferred oil-soluble corrosion inhibitor, can be added to your product at any point in your operation. Costs less than a tenth of a cent per barrel of product. Write our Products Department for detailed information. Ask about samples and how to test them.

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UNIVERSAL OIL PRODUCTS COMPANY

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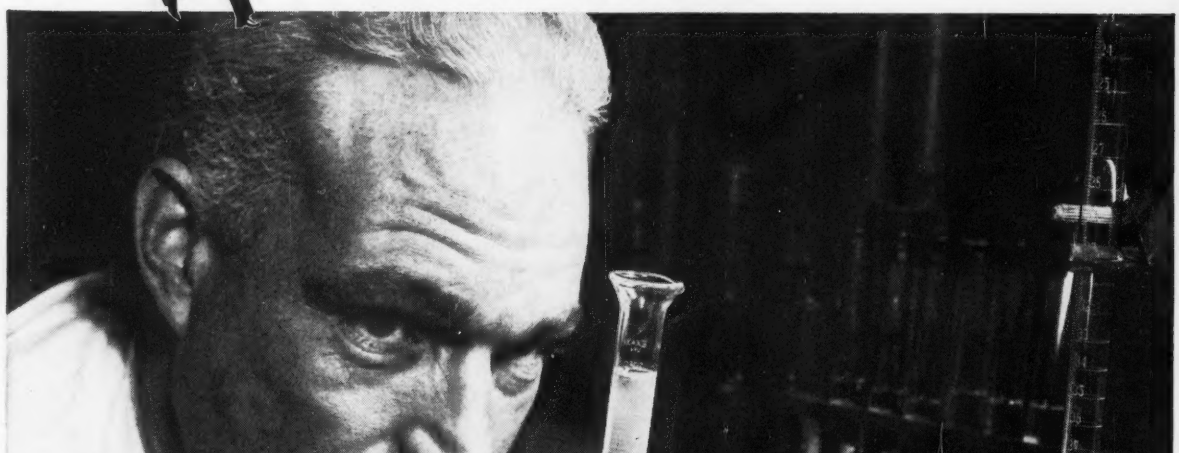
Des Plaines, Illinois, U.S.A.

More Than Forty Years Of Leadership In Petroleum Refining Technology

BRIDGEPORT "T" MEN* IN ACTION

**Technical Service Men*



The Case of the Acid Assailant

Where's the Culprit? Field spot tests of water samples give revealing clues as to cause of corrosion.

Case Solved. Initial recommendations go out to customers immediately and are followed by a detailed written report.

It was an ordinary afternoon in the Corrosion Lab when a sample of raw cooling water and a corroded condenser tube arrived from a midwest chemical company. Accompanying the evidence was a letter of explanation and a completed Technical Service corrosion questionnaire. The customer wanted help. Bridgeport "T" men swung into action.

The water sample was analyzed. It checked out against the original sample taken when Bridgeport tubes were first installed. Still no conclusive evidence of corrosive media was found in the water. Yet, examination of the tubes revealed them to be corroded both by acid and an abrasive.

The Bridgeport "T" Man visited the plant site and retraced the case step by step.

The first break came when a spot check was made of water taken directly from the cooling tower. A significant acid content was noted. Further on-the-spot examination showed that gas and fly ash from the plant smokestack were carried by prevailing winds over the cooling tower and deposited in large amounts in the tower. Closer examination of the cooling tower uncovered acid-forming sludge and abrasive fly ash in suspension.

The Bridgeport "T" Man, in an extensive report of his findings, recommended corrective measures. Company adopted recommendations. Tubes saved. Case closed.

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Behavior of Tin Alloys in Atmospheric Exposures*

Abstract

Results are reported from tests conducted at three sites to evaluate tin as a corrosion resistant coating for steel. Two marine and one heavy industrial atmospheric exposures were used in the tests. Deposits of two thicknesses of tin-zinc and tin-cadmium coatings are compared with equal thicknesses of zinc and cadmium. Comparative corrosion tests on tin-nickel and copper-nickel-chromium on steel at the three test sites are presented. Similar test data on bronze and copper as undercoatings for nickel and chromium are given. 2.2.5

TIN UNDERCOAT has been evaluated in a series of tests at Kure Beach, N. C., for marine atmospheric exposure and at East Chicago, Ind., for heavy industrial atmospheric exposure. In these tests, the basis metal was mild cold-rolled steel on which deposits of cadmium, zinc or tin under cadmium and tin under zinc were made. Plating thicknesses were 0.0003-inches. Where tin was used as an undercoat, it was deposited to 0.0001-inches and the zinc or cadmium to 0.0002-inches. Results of the exposures are shown in Table 1.

Several conclusions can be drawn from these findings. First, it is apparent that an undercoating of tin improves the corrosion resistance of zinc and cadmium plating in marine atmospheres. This improvement may be such as to make zinc plating over tin equal to straight cadmium plating for corrosion resistance in ocean areas. A tin undercoat for cadmium increases the corrosion resistance of that metal in salt air exposures, as shown in Table 1.

Results of exposure to heavy industrial atmospheres (not shown in Table 1) indicate that a tin undercoat has no beneficial effect on the resistance of either zinc or cadmium plating. The fact that test results were not the same in both locations indicates that the change in effectiveness is electrochemical rather than mechanical in nature.

Performance of organic coatings has been reported markedly improved by an application of an undercoating of tin, according to work done recently by the Tin Research Institute.[1,2] This work indicated that the amount of tin needed for this purpose is small enough to make its use feasible economically.

Tin Alloy Deposits

Tin-zinc, nominally co-deposited at a ratio of 78 percent tin and 22 percent zinc, is popular as a coating in cases where solderability is important. This alloy has the unusual feature of retaining solderability over long periods,[3] a desirable characteristic in coatings.

Another use suggested by the corrosion resistance in this alloy is application of the plating to another metal which is to be joined to aluminum. The tin-zinc plating markedly reduces the galvanic effect in these instances.

Tests with this plating in marine and industrial atmospheres indicate that it has lower corrosion resistance than cadmium in marine exposures and lower corrosion resistance than zinc in industrial atmospheres. This plating resists the formation of the so-called white rust better than does zinc.[4] Tin-zinc alloy is the only one of the three which has the solderability characteristic.

Another interesting feature of tin-zinc plating is its resistance to the so-called tin pest, the modification that tin undergoes on long exposure to extreme cold. Tests indicate that tin-zinc alloy plating probably can replace tin safely under these conditions.[5]

Tin-cadmium deposits plated in all proportions have been evaluated, but more research is needed. Tin and cadmium can be deposited separately and then alloyed through heat diffusion. This is the type of finish used to a limited degree in naval aircraft. Data indicate that this alloy, however deposited, has unusual resistance to corrosion caused by sea water.[6] However, these uses for the alloy are relatively minor.

Tin-lead alloys can be co-deposited through electroplating and research has indicated that the addition of tin to a lead deposit increases the corrosion resistance of the finish. This increase is maximum when tin co-deposited reaches 5 percent. Further additions make the corrosion resistance diminish.[7]

Tin-Nickel Deposits Useful

Tin-nickel, when co-deposited electrochemically, has a number of interesting characteristics. Tests indicated that this alloy is an intermetallic compound not found in the thermal equilibrium diagram and apparently cannot be made through the usual metallurgical methods. The throwing and covering powers of the bath, an acid electrolyte, are unusually good for this type of bath. The finish is solderable although the alloy is hard and almost bright. This makes it the only inexpensive decorative finish which has the solderability characteristic. Resistance of the alloy to many chemical reagents including acids and alkalis is high.[8]

Tests of this alloy, made with the cooperation of the International Nickel Company, have shown other facts. Comparative corrosion tests were run between tin-nickel and copper-nickel-chromium on steel in three atmospheres: Kure Beach, N. C., Pittsburgh, Pa. and Bayonne, N. J. The first was for marine exposure, the second for industrial and the third a combination of the two because Bayonne is near both salt water and heavy industrial activity.

TABLE 1—Tin as Undercoat for Zinc and Cadmium

Steel panels were exposed 80 feet from the ocean at Kure Beach, N. C.

10/1000 Inch	Material	% Area Rusted in Months Exposure			
		4	10	16	23
3	Zinc	e[1]	90	100	....
1 2	Tin Zinc	0	50[2]	60[2]	80[2]
3	Cadmium	0	15	60	90
1 2	Tin Cadmium	0	0	0	0

1. e = rust at edges and holes
2. average of 4 panels which were not consistent

Recording Systems Described

A note on the method of presenting the test data is necessary. The Kure Beach and Bayonne one-year inspections were recorded according to the standard ASTM system in which a number was assigned from 10 to 0 to indicate a gradient from a panel with no defects to one with more than 50 percent of the surface defective. Later data (Bayonne two-year and Pittsburgh) were given in two numbers, the first being a report on the protection of the basis metal and the second a report on the condition of the plating itself based on appearance, pitting, tarnish, etc.

The data as shown in Table 2 indicate that the marine atmosphere is more corrosive to both coatings than the industrial exposure. At Bayonne, after two years' exposure, tin-nickel showed superiority to nickel-chromium.

Tin-nickel panels fail differently than do nickel-chromium panels. The nickel-chromium plated panels fail through the formation of a number of small rust spots, indicating that corrosion takes place at the intersections of the minute cracks in ordinary chromium plating. Tin-nickel fails only at one or two spots with each spot growing larger as corrosion continues, indicating that the corrosion takes place only through pores already present in the metal. A flash of copper or bronze of about 0.01-mil was found to improve the performance of tin-nickel alloy deposits.

Tin-nickel retains its brightness in both marine and industrial atmospheres, but the slightly pinkish tone to the deposit increases with age, accentuating the difference between chromium's bluish color and tin-nickel's appearance. This would indicate that a thin chromium plate would be in order where the finishes are to be used close to each other.

(Continued on Page 114)

★ Extracted from a paper titled "Tin and Tin Alloys Offer Good Corrosion Resistance" by Robert T. Gore, Metal & Thermit Corp., New York 17.

Tin Alloys–

(Continued From Page 113)

Tin-Copper Alloys Tested

Tin-copper alloys can be plated in any proportion, but the two used most are the 40 to 45 percent tin alloy known as speculum and the 10 to 20 percent tin alloy called bronze.

Speculum usually is plated with a matte finish which has a high luster and resembles silver when polished. Although good for indoor use, speculum tarnishes quickly when used in outdoor atmospheres, especially those of industrial areas. Recent development of ways of depositing speculum bright has shown that the alloy will retain reflectivity better than silver or nickel in outdoor exposures.

Recently a series of tests was made on bronze plating similar to those on tin-nickel outlined above. The tests investigated the use of bronze and bright bronze as an undercoating for nickel and copper and also differences in the use of bronze and copper as undercoats for nickel and chromium.

Table 2 shows the results of the first series of tests. The nickel used was Watts' buffed; the chromium used was a conventional strike of about 0.000010-inch.

Table 3, with its data presented in the same manner as Table 2, shows the results of tests of bronze versus copper as undercoats for nickel and chromium. In these tests, the nickel was a proprietary bright nickel. The data show several interesting facts:

Bronze is superior to the same thickness of copper under nickel-chromium.

Bronze will permit using less than standard thicknesses of nickel without loss of corrosion resistance.

Bronze and chromium without nickel have good corrosion resistance, but this combination is not shown because the deposit deteriorates badly in appearance.

Although tin and tin alloys are not considered usually as coatings on steel to give corrosion resistance, a number of them show acceptable performance.

TABLE 2—Bronze and Bright Bronze Under Nickel and Copper

Lot	Plating (Thicknesses in Ten-Thousandths Inch)	RATINGS				
		Kure Beach		Bayonne		Pittsburgh
		6 Mo.	1 Yr.	1 Yr.	2 Yr.	28 Months
12	5 Cu + 5 Ni + Cr	5	2	2	2/0	8/6
21	5 Bz + 5 Ni + Cr	5	1	6	4/3	10/7
24	5 Brt Bz + 5 Ni + Cr	7	2	6	6/2	10/7
2	5 Cu + 5 Tin-Nickel	3	2	6	4/4	9/8
20	5 Bz + 5 Tin-Nickel	4	1	6	7/3	10/10
23	5 Brt Bz + 5 Tin-Nickel	3	0	7	7/5	10/10
14	10 Ni + Cr	9	6	2	1/0	7/6
27	10 Cu + Cr	0	2	0	0/0	0/0
19	10 Bz + Cr	8	9/1	0	7/0	8/1
22	10 Brt Bz + Cr	1	0	1	0/0	0/0
18	15 Ni + Cr	9	6	6	2/1	9/7
30	15 Cu + Cr	8	9/0	0	8/0	10/0
31	15 Bz + Cr	9	10/3	3	10/0	10/1
32	15 Brt Bz + Cr	9	1	8	9/2	10/8

TABLE 3—Bronze vs Copper Under Nickel/Chromium[1]

Lot	Plating (Thickness In Ten-Thousandths Inch)	Kure Beach 13 Months	East Chicago 13½ Months	Pittsburgh One Year
37	10 Cu + 2.5 Ni + Cr	9/7	8/0	8/2
49	10 Bz + 2.5 Ni + Cr	10/8	10/0	10/6
38	15 Cu + 2.5 Ni + Cr	9/7	9/0	8/2
50	15 Bz + 2.5 Ni + Cr	10/8	10/0	10/5
39	7.5 Cu + 5 Ni + Cr	8/6	9/2	10/6
51	7.5 Bz + 5 Ni + Cr	10/8	10/3	10/8
40	12.5 Cu + 5 Ni + Cr	10/8	9/0	10/8
52	12.5 Bz + 5 Ni + Cr	10/8	10/3	10/8
61	20 Cu + 2.5 Ni + Cr	9/7	9/0	10/4
62	20 Bz + 2.5 Ni + Cr	10/8	10/0	10/0
63	17.5 Cu + 5 Ni + Cr	9/7	9/0	8/4
64	17.5 Bz + 5 Ni + Cr	10/8	10/1	10/8

1. Nickel used is a proprietary (Unichrome) bright nickel.

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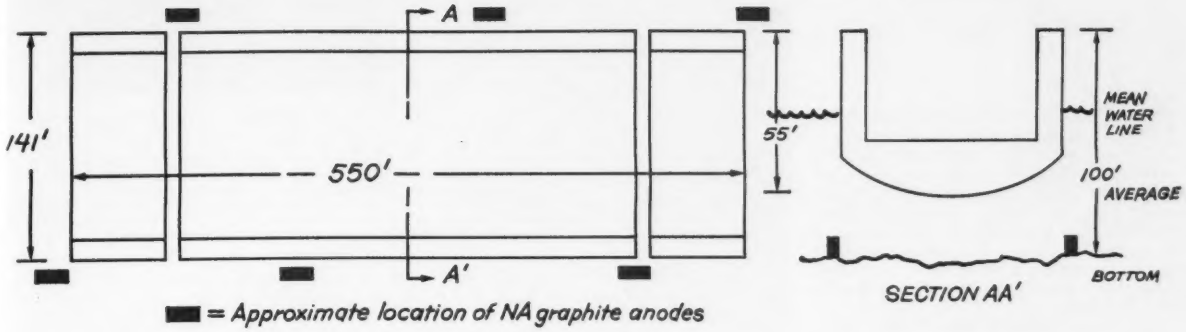

■ = Approximate location of NA graphite anodes

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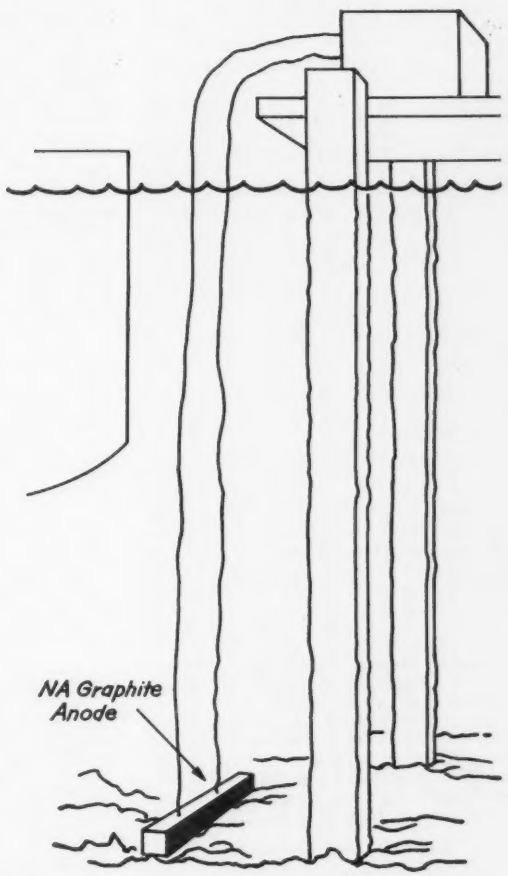
A corrosion engineer was given the problem of protecting a floating dry dock in a salt water harbor.

After a corrosion survey, the engineer decided to use impressed current cathodic protection and "National" NA graphite anodes for the submerged outside surface.

Six 12″ x 12″ x 72″ NA graphite anodes, located as shown above on the harbor bottom, are connected to the positive sides of six rectifiers. Each anode is discharging approximately 110 amps at 8.5 volts.

Large (approximately 600 lbs.) rectangular anodes were chosen to minimize movement on the bottom with tide currents. Two 125′ leads of 4/0 rubber insulated cable provide a low resistant pass between anodes and rectifiers.

The system has been in operation since 1955. Semi-annual inspections show complete protection with negative potential readings to a copper-copper sulphate reference cell greater than 0.85 volts.



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Improved Methods Plus Hot Spray

Speed Application of Saran Coatings*

Abstract

A saving of about one-half in application cost was achieved through efficiencies resulting from changed methods and hot spray application of Saran coatings to naval cargo tanks at Pearl Harbor Naval Shipyard. Also noted was substantially improved safety factors versus those for standard spraying method. New method reduced to one shift an operation formerly requiring two. 5.4.7

TWO PRINCIPAL changes were made in the methods used for applying Saran coatings at Pearl Harbor Naval Shipyard to bring the cost of application down to a low level while improving the safety factors. These changes were: 1. Modifying procedure in laying out tools and preparing material to give spray men more time in actual application, and 2. Adoption of 100 percent hot spray application methods.

Efficiencies resulting from these changes made it possible to do in one shift what formerly took two. At the same time significant progress was made in reducing hazards of working with Saran because man hours of exposure was reduced, numerous barrels of explosive and noxious solvents no longer are used and spray material was pumped directly to the heaters and pumps from dockside. Figure 1 illustrates gas concentrations measured during conventional and hot spray applications. Danger of spills and hazards from open buckets of Saran have been reduced.

One shift operation makes it possible to remove from the drydock at the end of the first shift all equipment capable of giving off hazardous vapors. Fresh water flushing of coated tanks further reduces concentrations of explosive vapor.

Spray Time Is Determined

The Painting and Sandblasting Shop analyzed lost time in the painting operation, including straining material, filling spray pot and other factors and learned that only three to three and one-half hours a shift actually was spent spraying. It was estimated that if this spraying time could be increased to six or six and one-half hours, a complete coating operation could be performed in an 8-hour shift.

Down time included preparing equipment, filling pressure pot, safety precautions, breaks, drying time between coats, changing material, equipment failure, waiting for inspection of coat, high gas reading delays, lunch hour and end-of-shift time (when there is insufficient time to finish a new coat); cleaning up equipment, removing and storing safety clothing and equipment and personal cleanup time.

Radical Changes Indicated

In order to reduce to one-shift operation what formerly had been a two-shift application several radical changes were necessary, the most important of which was scheduling work so production personnel would be able to start spraying immediately upon reaching the job site. This was accomplished by having equipment and materials ready, even to the extent that the spraymen's lines, guns and breathing air lines were in the tank ready to operate when they arrived.

Ventilation was installed and tested before starting time for the spray work and safety clearance obtained so there was no delay.

Spray time was further extended by scheduling all clean-up time on the shift following the actual coating operation. The production sprayman leaves equipment removal and cleanup of mixers, machines and pumps to a few men on the next shift who do the work at the shop. This means there is no interference with craftsmen who work on the next shift.

The efficiencies inherent in hot spray application were essential if sufficient thickness was to be applied in the allotted time. Furthermore, because materials used in hot spray application are pumped directly to the gun from the mixing tank, the need to strain materials and load conventional pressure vessels was removed. These all resulted in more efficient and continuous production. Drying time was no problem because the three or four coats applied dried quickly.

Because men doing the work need to understand the reasons for changes in procedures and methods, once-a-week, one-hour training sessions were held for the small crew that does Saran and vinyl work. Operation of the hot-spray equipment,* advantages of application and the need for a reduction in application time was explained. Employees cooperated fully and assisted by making suggestions leading to reduction in stand-by-time. They are proud that they are now doing a better job at lower cost.

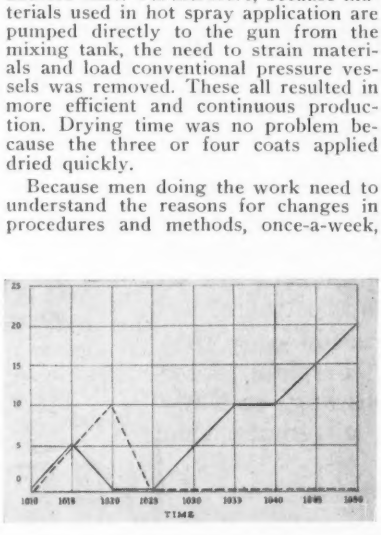

Figure 1—Gas readings during spraying of Saran by hot spray compared to readings taken using conventional methods on U. S. S. Pickerel. Conventional spray shown by solid line; hot spray by broken line.

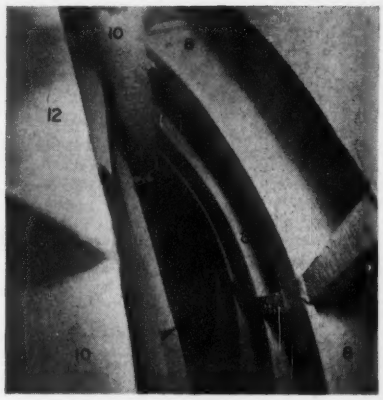

Figure 2—Uniformity of Saran coating thickness using hot spray method is shown in this photograph of the starboard safety tank of the U. S. S. Pickerel where gauged dry thickness has been indicated in mils for various parts of the tank. Saran was sprayed at 4-to-1 solvent-to-pigment ratio at 140 F, air 45 psi, fluid presure 45 lb. to obtain mil thicknesses as follows: One Pass—1¾ mils; two passes 4 mils; three passes, 6 mils.

Cost Record Is Kept

A record was made of costs on coating with Saran four cargo tanks in the YOG 78. The job required 22 painters and three gas testers. The final cost as indicated in Table 1 was 17.7¢ per

★ Condensed from data supplied by Harland A. Morley, Master Painter, Pearl Harbor Naval Shipyard, Honolulu, T. H.

* The yard uses five electrically heated Speed-Flo units serving 15 guns and other steam-heated units.

(Continued on Page 117)

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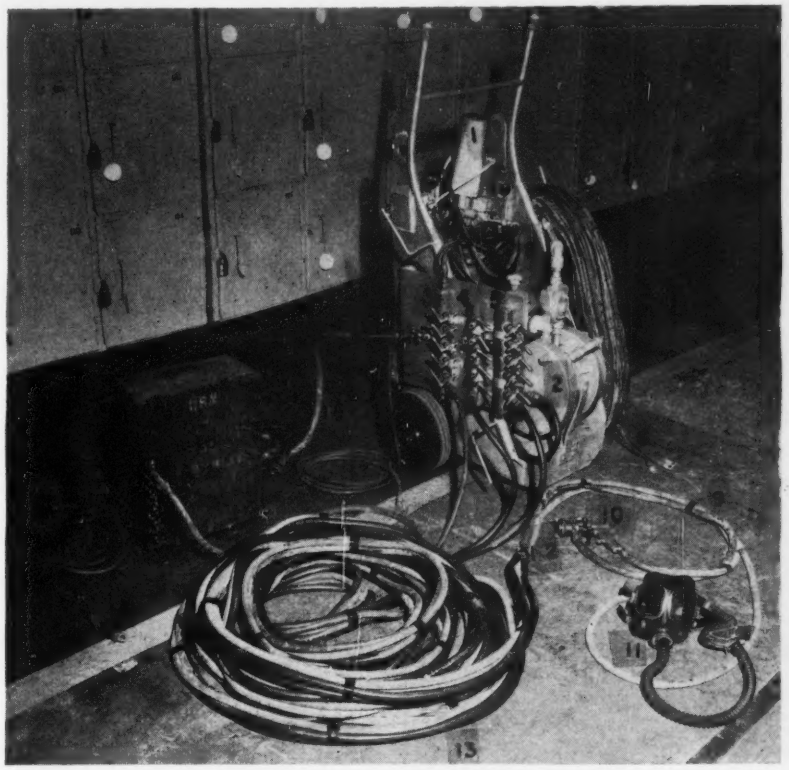
Hot Sprayed Vinyl Saves 20% on Ship Hulls

Tests at the Pearl Harbor Naval Shipyard have shown that there is a saving of about 20 percent in the cost of hot sprayed vinyl coatings on hulls of Navy ships versus conventional hot plastic systems. The savings come from extended life expectancy of the vinyl system, fuel and speed advantages and because of faster application with hot spray.

Records at the shipyard indicate hot vinyl systems will cost 13 cents a square foot less than the conventional hot plastic system. Because of greater application speed, a net saving of 21 cents a square foot versus hot plastic systems is indicated.

Future estimates of underwater coatings at the shipyard, it was reported, will be based on the hot vinyl system.



The machine shown is a Navy modification of the paint heater-pump system made by Spee-Flo Corp., Houston. This is called a 4-gun unit by the Navy and was developed at Pearl Harbor Naval Shipyard. Essential parts of the unit as indicated by numbers are as follows: 1) Pump on tank (to pump material to heater). 2) Paint heater on cart. 3) Atomizing air manifolds (for spray guns). 4) Hot paint return manifold (returns to tank). 5) Hot paint out-flow manifold from heater. 6) 200-foot coil electric cord. 7) Thermostat. 8) Watch face thermometer. 9) Short piece single hot paint line (for flexibility. 10) Copper-coated (anti-spark) spray gun. 11) Breather mask. 12) Hot paint return yoke (away from gun). 13) 75-foot coil hot paint line, breather and atomizing air lines (forms single unit). 14) Grounding wire for breather filter box (static). 15) Breather filter box (to purify air). 16) Main air supply line (90-lb). 17) Grounding wire for cart. 18) Lines from manifold to tank top (air for pump, paint lines to heater and from return manifold). 19) Agitator.

Saran Coatings—

(Continued From Page 116)

square foot for application of an average of 8 mils, dry thickness, during one shift. This was less than half the cost based on conventional application of a similar thickness on eight tanks.

This work was done with equipment used by the yard for hot spray application of vinyls, Saran and other materials.

TABLE 1—Cost of Saran Coating by Hot Spray

ITEM	Quantity	Average Rate/Day or Unit Cost	Total
Saran Material..	695 gal.	$ 1.90	$1320.50
Methyl Ethyl Ketone.....	110 gal.[1]	$ 1.00	110.00
Men Required..	25	$48.00	1200.00
Total Cost.....	.....	.....	$2630.50
Total Square Feet.....	14,860		
Cost Square Feet	$.177[2]		

[1] For cleanup and flushing equipment only.
[2] Dry film thickness, average 8 mils.

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A new Division on Materials Sciences is being organized by ASTM to intensify development of knowledge of the fundamentals of materials. It will be concerned with the collection, establishment and publication of basic information essential for a better understanding of materials and their properties.

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Bridge Caulking Technique Helps Solve Aerial Pipeline Crossing Problem*

Abstract

Bridge caulking techniques are discussed as a means of solving corrosion problems of pipeline aerial crossings. Application techniques are outlined. Methods of preventing corrosion around pipeline hangers and foundation bolts are discussed, and periodic inspections are recommended.

5.4.5

BRIDGE CAULKING techniques are being used to solve the coating problem in aerial pipeline crossings. Caulking of all cracks and crevices common to riveted structures and sealing of back-to-back angles help make the coverage of a protective coating on these aerial crossings almost perfect.

Application of caulking material after the prime coat and before the finish coats also improves the structure's appearance. Catalyzed epoxy materials made up of 100 percent solids are effective caulking compounds if the correct formulation is obtained to give good working characteristics. Too thin a material tends to sag on vertical seams and to drip from overhead applications. Too stiff a material is difficult to apply and consequently expensive because of increased labor costs. Caulking materials with good work ability must be stiff enough to seal a ½-inch gap between back-to-back angles without completely filling the space between angles. A pot life of 30 to 60 minutes for the resin formulation gives quick hardening and reduces possible damage to completed work. All the necessary conditions of a catalyzed epoxy caulking compound can be obtained through cooperation with the distributor.

Three pipeline bridges for Transcontinental Gas Pipe Line Corporation have been caulked in conjunction with paint jobs, and a fourth is planned. Experience gained as new problems in bridge caulking were encountered and solved made each job progressively more satisfactory.

To determine if plates and seams could be sealed satisfactorily to improve structure protection, an original test was made on the Atchafalaya River Bridge one year before the bridge was scheduled to be sandblasted and painted. If the test proved unsatisfactory, the material was to be removed when the structure was sandblasted for re-painting. The test material was removed after an analysis was made. A complete caulking job was applied in conjunction with the planned painting job. Recently inspected, this caulking job has shown no visible coating or caulking failure after 1½ years.

Pipeline hangers also need attention to prevent corrosion between the pipe and hangers where moisture can be trapped. Application of glass fiber and cold mastic to the contact area between pipe and hanger has proved to be flexible enough to withstand some movement and still seal the contact area for protection. The same mastic will coat poorly galvanized cable clamps and fill all clamp voids to prevent entrapment of moisture.

Complete protection of foundation bolts, a structural feature of bridges, is difficult to obtain. These bolts are usually in such a position that the back side of the bolt and the structure behind it cannot be cleaned or painted well enough to prevent corrosion.

Solution to this problem was obtained by enclosing the structure where the bolts enter the tower base. One-quarter inch pipe couplings were welded into holes cut into the top and bottom of the box formed around the foundation bolt. Cold mastic was pumped into the bottom coupling until the chamber was filled and mastic was showing at the top coupling. Severe corrosion of foundation bolts has been controlled by this method.

After a paint film has been applied on a well cleaned structure and after the weak points in the film have been protected by caulking, periodic inspections should be established to maintain protection conditions.

The first inspection should be within the first year, preferably six months after completion of the painting. Coating holidays should be touched up within the first year. Then yearly inspections should be given until a maintenance coat is needed.

Time between the original painting and maintenance applications can range from three to six years depending on materials used and atmospheric conditions at the structure.

★ Extracted from a paper titled "Corrosion Control on Aerial Pipe Line Crossings" by Gordon Davis, Transcontinental Gas Pipe Line Corp., Houston, presented at a meeting of the South Central Region, National Association of Corrosion Engineers, October 20-24, 1958, New Orleans.

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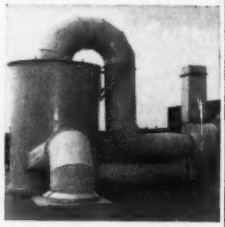

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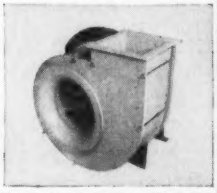

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New Formulations Give Versatility to Urethane Coatings, by Barnard Goodman

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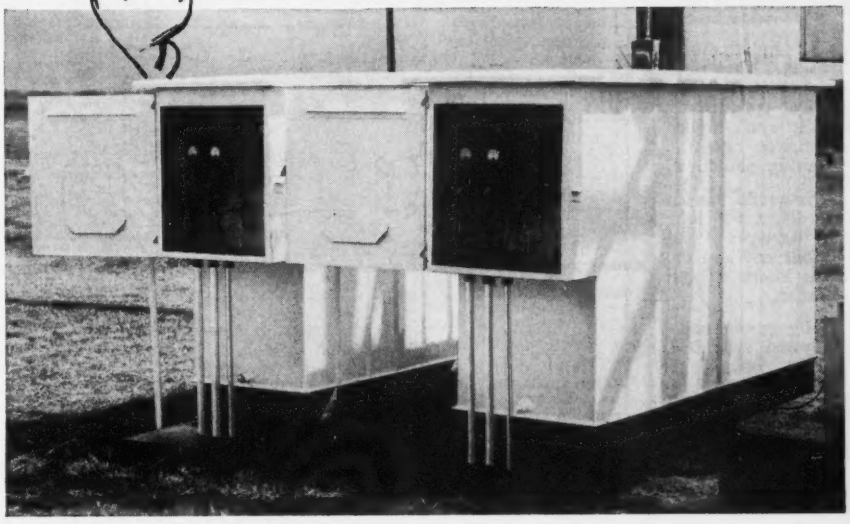

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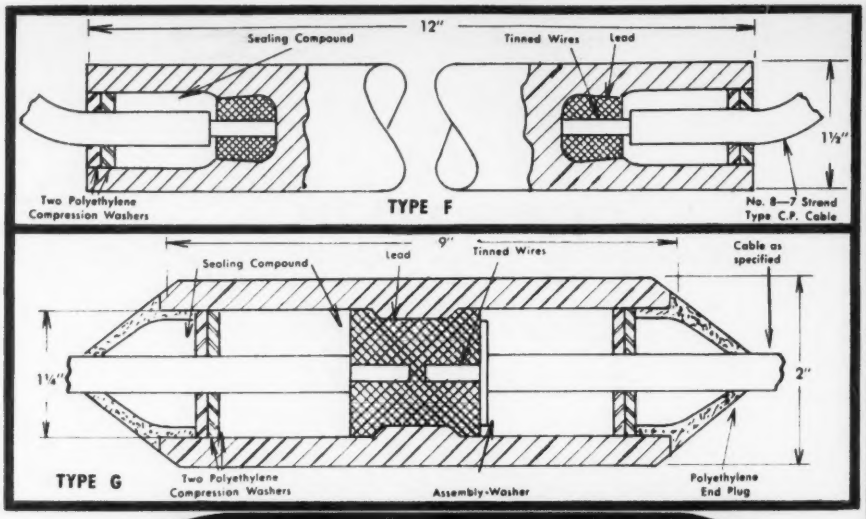

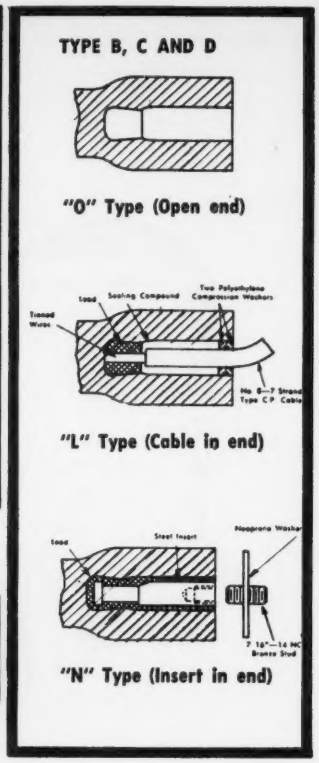

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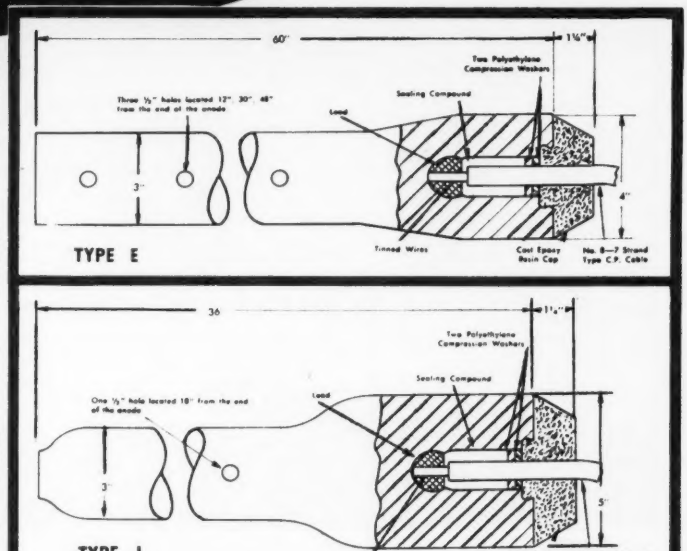

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CORROSION ABSTRACTS

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3. CHARACTERISTIC CORROSION PHENOMENA

3.6 Electrochemical Effects

3.6.5, 3.4.7

Effect of pH on Corrosion Potentials. I. A. Ammar and S. Riad. J. Phys. Chem., **62,** No. 2, 150-154 (1958) Feb.

The dependence of the potential of a corroding metal on the pH of solution has been considered. This has been done by considering the recent aspects of the kinetics of hydrogen evolution and metal dissolution, and expressions for the corrosion potential, E_c, have been deduced under the condition when the reverse rates for the cathodic and the anodic reactions can be neglected. The expressions derived for (dE_c/dpH) have been tabulated. The effect of oxygen on corrosion potentials has also been studied by considering the cathodic reaction, in the presence of oxygen, to be the reduction of oxygen by discharged hydrogen. (auth.)—ALL. 15448

3.6.8

Hydrogen Overpotential on Electrodeposited Nickel in Sodium Hydroxide Solutions. I. A. Ammar and S. A. Awad. J. Electrochem. Soc., **104,** No. 11, 686-690 (1957).

Hydrogen overpotential on electrodeposited nickel in 0.01-1.0 N-sodium hydroxide was measured from 25 to 55 C. The heat of activation at 25 C at the reversable potential was 8.8 ± 0.1 k.cal./mole and the effect of pH on η was calculated to be 26 mV/unit increase pH—much $<$ the theoretical value. From measurements at very low current density the electron number was observed to be very near unity and the Tafel slope increased with temperature. The rate-determining step is represented by $H_2O + e \rightarrow OH^- + MH$, where M is the electrode surface. The results were compared with those obtained from measurements on nickel wire. 21 references.—MA. 15552

3.6.8, 3.7.4, 6.3.3

The Influence of Crystal Structure on the Hydrogen Overvoltage of Chromium. St. G. Christov and N. A. Pangarov. Z. Elektrochem., **61,** No. 1, 113-121 (1957).

Chromium was deposited from a bath containing 350 g/l chromium trioxide, 3.5 g/l sulfuric acid, at 0.35 amp/cm² and 45 C and hexagonal chromium from 0.075 normal sulfuric acid, 600 g/l chromium trioxide, 32.4 g/l sugar at 0.16 amp/cm² and 18.5 C. Conditions for best reproducibility of overvoltage measurements were established. Not only the mechanism but also the value of the overvoltage differed for the two crystal forms. The differences are explained by the different adsorption characteristics of the cubic and hexagonal crystals. 34 references.—MA. 15779

3.6.5, 6.2.2

Electrode Potential Studies on Iron in Dilute Phosphate-Chromate Solutions. R. N. Ride. J. Applied Chem., **8,** Pt. 3, 175-183 (1958) March.

Electrode potential behavior of iron in aerated potassium phosphate (2500 ppm) is reproducible at pH 5.2 and 6.1, major breakdown and removal of pre-immersion oxide film being followed by slow, uniform corrosion. When potassium dichromate (30 ppm) is subsequently added, potential rises logarithmically from a critical, pH-dependent value and re-passivation due to dissolved oxygen alone is a much slower process. Potential behavior is not reproducible in solutions which contain both phosphate and chromate from the start. Generally, pre-immersion oxide is not removed but maintenance of passive state is unpredictable since it depends on capacity of inhibitive system to block effectively sporadic pores in film with precipitated hydroxide. Significance of potential values was examined in relation to pH-potential equilibrium data for iron-water system. Reasonable agreement between breakdown potentials and theoretical values was observed. Graphs, 21 references.—INCO. 15649

3.6.6, 3.2.2, 7.7

Solid-State Dissolution of Germanium by Indium in Semiconductor Devices. John Roschen and C. G. Thornton. J. Applied Physics, **29,** 923-928 (1958) June.

The development of small pits or cavities was observed in germanium semiconductor devices with indium metal to germanium semiconductor contacts at temperatures below the melting point of indium. The mechanism of cavity formation is given as well as a method of prevention of solution cavities.—BTR. 16108

3.6.6, 6.4.2

Use of Aluminum in Contact with Other Metals. (In Italian.) A. Prati. *Ingegneria Meccanica,* **6,** 9-16 (1957) July.

Corrosion tendencies of more common assemblies of aluminum and other metal were evaluated and order of preference to be followed in selection of components of such bimetallic assemblies was established. 6 references.—MR. 14991

3.6.9

Corrosion by Stray Currents. (In Italian.) T. A. Turco. *Pitture e Vernici,* 13, 589-592 (1957) Sept.

Abstracts in This Section are selected from among those supplied to subscribers to the NACE Abstract Punch Card Service. Persons who are interested in reviewing all available abstracts should write to NACE for information on this service.

Abstract Pagination Printed Incorrectly For Jan-Feb Issues

Pages of the Corrosion Abstracts section in the January and February issues were numbered incorrectly in the lower outside margins where the "a" series numbers are carried. The January issue should have begun with 1a.

Corrected numbers will be printed on the errata page in the December, 1959, issue of CORROSION.

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Mechanism of corrosion by stray currents and newer methods of combatting same, including electrical measures and coatings of various types. 8 references. —MR. 15098

3.6.6, 6.6.4

The Compatibility of a Number of Metals and Alloys with Graphite. Arnold F. Gerds and Manley W. Mallett. Battelle Memorial Inst. U. S. Atomic Energy Commission Pubn., BMI-1261, April 14, 1958, 30 pp. Available from Office of Technical Services, Washington, D. C.

The interaction between graphite and a number of metals and alloys was studied at 1850 F in 250-hr tests. Some materials were also tested at 1650 and 1850 F in 1000-hr tests. The graphite was heated in intimate contact with the test metal in welded metallic capsules. The extent of the reaction was judged on the basis of metallographic examinations and hardness traverses on cross sections of the reacted elements. Copper was the least reactive material tested in contact with graphite. Nickel was only slightly more reactive. Outside of a very hard carbide band at the graphite-metal interface, no evidence of interaction of carbon with molybdenum was found. Inconel X was the most compatible alloy tested in contact with graphite, although some pickup was observed. Alnicro was the next best alloy. Nichrome V, Inconel, and Types 316 and 318 stainless steels were all much more reactive. Copper and chromium plating reduced the rate of diffusion of carbon into Type 316 stainless steel. Nickel plating was less effective for this purpose. (auth)—NSA. 15951

3.6.8, 6.4.2

Hydrogen Evolution from Duralumin Exposed to Acid Salt Solution. T. Marshall and G. J. Schafer. J. Applied Chem., **8,** Pt. 5, 303-310 (1958) May.

The evolution of gas from duralumin corroding in an aqueous solution containing 10 g of hydrochloric acid plus 30 g of sodium chloride per liter of solution was studied. To obtain reproducible gas-evolutions it was found necessary to control specimen surface pretreatment and the temperature and composition of the corrosion solution. Duralumin which corroded by pitting evolved gas at an approximate constant rate after an induction period of several hours. The same duralumin, heat-treated so that it was susceptible to intergranular attack, evolved gas at a rate increasing linearly with time of immersion, after a short induction period. —BTR. 16063

3.6.8, 4.3.2, 6.2.3, 6.2.5

The Corrosion of Stainless and Carbon Steels During Cathodic Polarization in Nitric Acid Solutions. (In Russian.) N. D. Tomashov, M. M. Kurtepov and E. N. Miroliubov. J. Phys. Chem., USSR (Zh. Fiz. Khim.), **32,** No. 4, 904-908 (1958) April.

Study of possibility of partial or complete breakdown of the passive state and the subsequent development of cathodic protection of stainless and carbon steels in 3% nitric acid solutions. Steels IX18H11B and 3 were investigated. The dependence of the corrosion rate on the acid concentration, temperature, and duration of polarization of these steels polarized cathodically to a given potential is similar to that for carbon steel without polarization.—BTR. 16088

3.6.8, 6.4.2

Polarization Characteristics of Aluminum and Its Alloys. (In Japanese.) Goro Ito. Light Metals, **8,** 93-106 (1958) Jan.

Polarization in chloride solution shows that the cathodic reaction is the rate-determining step and depolarization of the cathode increases the corrosion of the anode. With two dissimilar metals in contact in electrolytic solution, the corrosion current is not dependent on the open circuit potential difference but on the polarization characteristics of the couple. With aluminum-copper couple in chloride solution, copper polarizes in a more marked degree than aluminum and the cathode controls the corrosion reaction. Contact corrosion of aluminum or various metals with other metals can be inhibited by external emf and the potential break in cathodic polarization curve for these couples can serve as a criterion of protection. 15 references.—MR. 15577

3.6.5, 3.6.6

The Electrochemical Series of Metals in Common Corroding Media. J. Elze and G. Oelsner. Metalloberflache, **12,** 129-133 (1958) May.

Investigation to establish whether it is possible to derive electrochemical series of metals in common corroding media and whether the differences in potential between two metals in the series can be related to galvanic and other corrosion effects. Determination of electrode potentials, by technique detailed, in water and sea water: data are included for inter alia, nickel silver, Monel, brass, K Monel, nickel-beryllium alloy, 18/8 steel, carbonyl iron, etc.: compositions of all materials tabulated.—INCO. 15946

3.6.6, 6.2.5, 6.4.2

Investigation of the Galvanic Corrosion of 304 Stainless Steel, 2S Aluminum and 72S Aluminum. Wayne W. Binger. Chicago University. U. S. Atomic Energy Commission Pubn., CT-3030, June 2, 1945 (Declassified Feb. 16, 1957), 28 pp. Available from Office of Technical Services, Washington, D. C.

Electrochemical data were obtained in a flowing system on the galvanic corrosion of 304 stainless steel, 2S aluminum and 72S aluminum couples, and these data are correlated with data obtained in stirred beaker tests under similar conditions in simulated river water.—NSA. 15728

3.6.2, 4.6.2, 6.3.17

Crevice Corrosion of Uranium and Uranium Alloys. J. W. Frank and A. H. Roebuck. Argonne National Lab. U. S. Atomic Energy Commission Pubn., ANL-5380, March, 1955 (Declassified March 1, 1957), 39 pp. Available from Office of Technical Services, Washington, D. C.

Three types of experiments were undertaken in order to study the corrosion of uranium and uranium alloys in high-temperature (500 C) water. Results of the capsule and bonded plate-pin hole experiments showed that corrosion of enclosed uranium is markedly different from that of uranium in open contact with water. The gaseous hydriding experiments showed a definite relationship between rate of reaction and temperature, gas pressure and alloy reacting. The corrosion of uranium in lithium hydroxide solutions was also studied.—NSA. 14926

3.6.2, 6.2.5

On the Mechanism of Corrosion of Metals in Narrow Slits. Pt. IV. The Corrosion of Stainless Steel. (In Russian.) I. K. Marshakov and I. L. Rozenfeld. Zh. Fiz. Khim., **32,** No. 1, 66-72 (1958) Jan.

The corrosion and electrochemical behavior in narrow cracks and crevices in 0.5 Normal sodium chloride solutions of X28, X17, 2X13, and NL steels with 3% chromium and low carbon steel. The corrosion of the stainless steel in the crevice in contrast with that in the bulk of the electrolyte increases greatly owing to the formation of active-passive cells the anode of which is the metal in the crevice.—BTR. 16000

3.6.5

Potential Fluctuations of Iron and Zinc Electrodes in Sulphuric Acid. Hans Ludering. Arch Eisenhüttenw, **28,** No. 10, 625-632 (1957).

Fluctuations in potential occur when the current density/potential curve decreases more steeply than the straight line for ohmic resistance at the point of intersection. However, there are some points on the current density/potential curves for iron and zinc in sulfuric acid where fluctuations occur although the current density curve is horizontal. All fluctuations indicate that energy is stored; in the present case in the condenser formed by the metal surface and the double layer film. If the charge on this condenser decreases with rising potential, fluctuations may occur. In the case of iron and zinc in sulfuric acid in the potential range where fluctuations occur, the charge d/voltage curve is N-shaped. A method for determining this curve is described. The peculiar shape of the curve is due to a diminution in the amount of material in the double layer in the relevant potential range. Conditions for electrode stability are discussed.—MA. 15823

3.6.6, 4.3.2

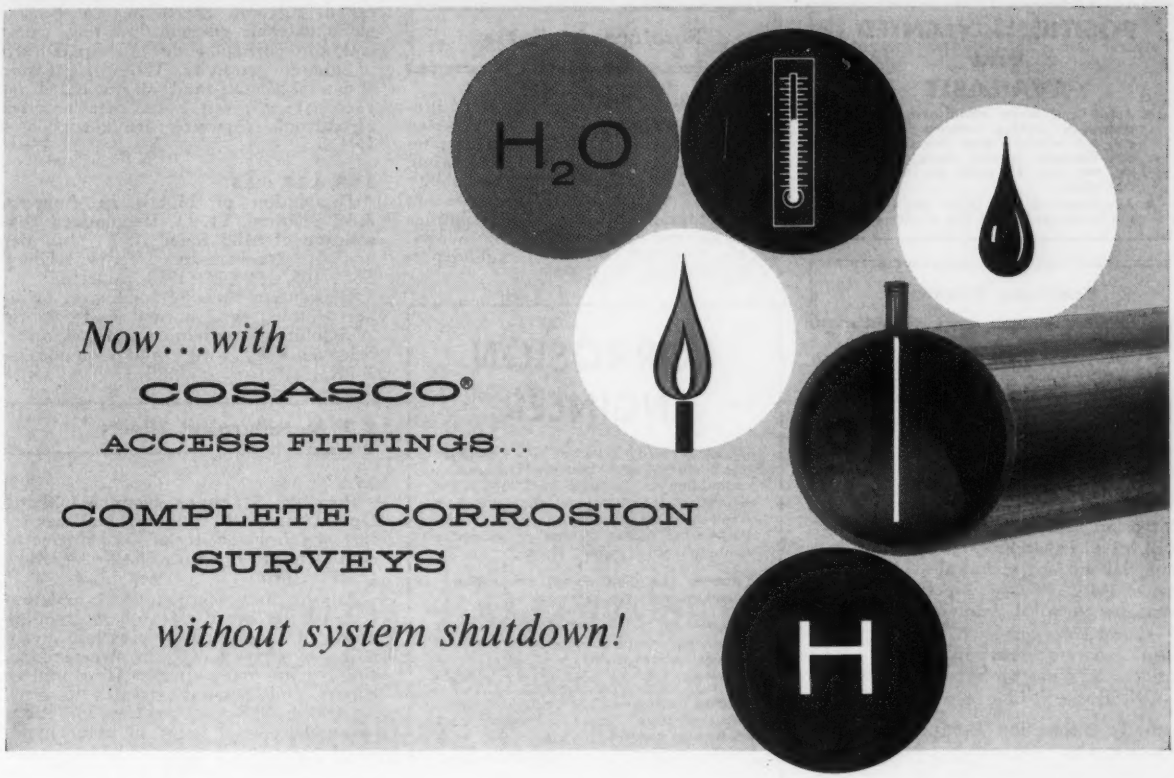
Corrosion of Ten Metals in Boiling Hydrochloric Acid When in Contact with Rhodium, Palladium, Iridium and Platinum. W. R. Buck and H. Leidheiser. Nature, **181,** No. 4624, 1681-1682 (1958) June 14.

The corrosion of 10 metals, including zinc and cadmium, in boiling 2M hydrochloric acid was studied both alone and in contact with platinum, palladium, iridium and rhodium. For all the metals except titanium the rate of corrosion was increased when in contact with one of the platinum metals, and the relative effectiveness of the platinum metals in increasing corrosion varied with the metal corroding. For zinc, cadmium and six other metals the average rate of corrosion when coupled with a platinum metal was related to the standard electrode potential.—ZDA. 16058

3.6.6

Corrosion Experiences with Dissimilar Metals. A. A. Brouwer. Dow Chemical Co. Ind. and Eng. Chem., **50,** No. 4, 73A-74A (1958) April.

When dissimilar metals in electrical contact are exposed to a corrosive or conductive medium, galvanic current flows from 1 metal to the other. Corrosion of the more active or anodic member of a couple usually increases by an amount directly related to the galvanic current flow by Faraday's law. This portion of the total anode corrosion is



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called galvanic corrosion. In certain cases galvanic current flow may cause cathodic corrosion due to breakdown of inherent passivity. Table of galvanic series of metals and alloys based on Inco data is given. Some case histories of galvanic corrosion are described.—INCO. 15609

3.6.8, 4.3.2, 6.2.3

Dissolution of Metals in Aqueous Acid Solution. Pt. II. Depolarized Dissolution of Mild Steel. A. C. Makrides and N. Hackerman. J. Electrochem. Soc., **105,** 156-162 (1958) March.

Dissolution rates of mild steel cylinders rotated in 2 normal hydrochloric acid solutions containing ferric chloride, benzoquinone, and tolu-p-quinone.—BTR. 15658

3.7 Metallurgical Effects

3.7.3

Residual Stresses from Machining Operations. E. K. HENRIKSEN. Paper before American Society of Tool Engineers, Ann. Mtg. *Tool Engr.,* **38,** No. 4, 92-96 (1957) April.

Author discusses own work and that reported by other researchers covering origin of machining stresses and mechanism of stress formation, stresses from single-point tools, stresses from milling cutters and grinding stresses in steel. Austenitic 18-8 has capacity for stress accumulation not found in other materials; when cut is repeated under identical conditions, second cut results in higher residual stress than first, stress value levels off after number of cuts and then decreases slightly. This behavior is attributed to work-hardening. Among graphs is one showing milling stresses induced in 4340.—INCO. 14069

3.7.3

Nickel-Chromium-Boron Brazing of a High-Temperature Alloy. G. Aggen, R. A. Long and E. E. Reynolds. Paper before Am. Welding Soc., Nat'l. Spring Mtg., Philadelphia, April 8-12, 1957. Welding J., **36,** No. 8, 366s-372s (1957) August.

Brazing investigation conducted on sheet and bar specimens of A-286. Brazing was performed in continuous-belt furnace under dry hydrogen atmosphere. Nickel-chromium-boron brazing alloy meeting AMS-4775 specifications was used in joining all specimens. Descriptions are given of brazing furnace and brazing cycles. Although A-286 contains 2 titanium and 0.2 aluminum, it was not necessary to plate surfaces in order to obtain satisfactory joints (contrary to previously published results). Method of surface preparation is described. Tests on brazed joints include smooth- and notched-bar rupture tests at 1200 F and short-time tensile tests at room temperature, 1200 and 1300 F. Metallographic examinations were made to determine effects of brazing alloy and brazing time-temperature cycles on microstructure of base alloy. Three types of specimens evaluated were: surface-brazed specimens, butt-brazed bar specimens and double-lap sheet specimens. Tables, photomicrographs.—INCO. 16123

3.7.3

Solder Bonding Aluminum to Uranium. H. W. Mishler, J. T. Niemann, R. P. Sopher and D. C. Martin. Battelle Memorial Inst. U. S. Atomic Energy Com-

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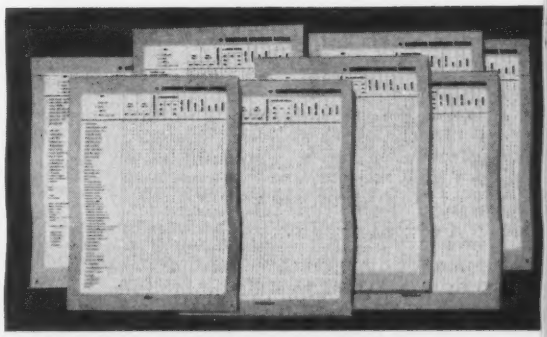

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mission Pubn., BMI-923, July 7, 1954 (Declassified Feb. 26, 1957), 28 pp. Available from Office of Technical Services, Washington, D. C.

This study was undertaken to determine if flat-plate fuel elements could be produced by solder bonding aluminum sheets to a uranium core. It was necessary to plate the uranium with chromium, iron or nickel, which served as a diffusion barrier. The solders used were lead, tin, zinc and their alloys. No provision was made for removing surface oxides on the base metals. Therefore it was necessary to plate the aluminum and the uranium diffusion barrier. The best bonds were produced with aluminum plated with 0.5 mil of copper and nickel-plated uranium given a copper flash. Corrosion tests on bonded samples showed zinc was the most corrosion-resistant solder tested. Successive samples made with like solders sometimes varied in joint quality. A greater variation would probably exist in larger samples made under mass-production conditions. (auth.)—NSA. 15869

3.7.3

Mechanical Properties of Welded Titanium Joints. Karl Bungardt and Klaus Rüdinger. Z. Metallkunde, **48,** No. 6, 335-340 (1957).

Specimens of two thicknesses of sheet of two compositions were A-arc welded using filler rod of similar composition. In the seam the UTS and PS are higher, and the elongation, reduction in area, notch-impact and fatigue strengths considerably lower than in the regions unaffected by heat; thick sheet shows greater differences than thin sheet; annealing at 700 C for 1 hr tends to reduce the variations. The cold deformability of the sheet, as evidenced by rolling parallel and perpendicular to the seam direction is unaffected by welding, as is the corrosion-resistance to 3 and 5 percent hydrochloric acid at 20 C, boiling 2 percent hydrochloric acid, and boiling 10 percent sodium hydroxide. Gas pick-up during welding is small.—MA. 15520

3.7.3.

Studies of Upset Variables in the Flash Welding of Steels. E. F. Nippes, W. F. Savage, G. Grotke and S. M. Robelotto. Rensselaer Polytechnic Inst. *Welding J.,* **36,** No. 4, 192s-216s (1957) April.

Objectives of test were to determine effects of temperature distribution at the instant of upset and upset current density on upsetting action; to study effect of upset distance on microstructure and mechanical properties of flash welds; and to investigate by means of high-speed photographic and oscillographic means the influence of changes in secondary reactance of welding transformer on flashing behavior. Flash welds were prepared in AISI 1020, 4130 and 4340 steels, employing 2 rates of platen acceleration and controlled amounts of current density. Tests consisted of bending to initial failure, full guided-bend testing and U bend testing. Significant differences were noted in the three steels in nature and severity of cracking, crack location and in incidence of flat-spots and weld-line cracks. Macrographs of similar welds show effect of variations in upset distance and platen acceleration on orientation of fiber structure in vicinity of joints. Hypothesis for formation of flat-spots is presented. Numerous tables, graphs, photomacrographs.—INCO. 14147

3.7.3

Ultrasonic Welding—a New Technique Grows. J. B. Jones. Paper before Am. Welding Soc. and Am. Soc. for Metals., Washington, Dec. 9, 1957. Metal Progress, **73,** No. 4, 68-72 (1958) April.

Ultrasonic welding unites similar or dissimilar metals by introducing vibrational energy into the area to be joined. Solid-state metallurgical bonds, comparable in strength to those resulting from conventional methods, are produced. Thin gages are readily welded, even in high-temperature, corrosion resistant metals. Metals welded by this method include several types of stainless steels, molybdenum, zirconium, Inconel, Monel and tantalum. Photomicrograph shows ultrasonic welded bond between nickel and gold-plated Kovar. Tables, graphs, photos.—INCO. 15645

3.8 Miscellaneous Principles

3.8.4

Chemical and Electrochemical Affinities of Corrosion Processes. Pierre Van Rysselberghe. Stanford University. Corrosion Technology, **5,** No. 2, 49-52, 62 (1958) Feb.

Analysis of corrosion reactions made possible by use of concepts of chemical and electrochemical affinities, principles of equilibrium thermodynamics and irreversible processes. Whole presentation is based on a typical example, that of metallic zinc undergoing corrosion in aqueous media. Generalization to other cases can readily be worked out. In considering metal dipping freely in aqueous solution, effects of added acid, oxygen at saturation and corrosion inhibitor in solution are discussed. Section dealing with metal dipping in aqueous solution and with applied current covers lowering of electrical potential (cathodic protection), current corresponding to single electrode process, simultaneous electrode processes in neighborhood of equilibrium and coupling of electrode processes (states of minimum entropy production).—INCO. 15579

3.8.4

Uncommon Valency Ions and the Difference Effect. M. E. Straumanis. J. Electrochem. Soc., **105,** No. 5, 284-286 (1958) May.

It is shown that in evaluating the rates of anodic dissolution processes it is necessary to consider the influence of the positive and negative difference effects; otherwise ions going into solution with uncommon valency may be found. Scale fragments detached from an aluminum anode by the current passing through it could be seen. The hypothesis of valence change of ions while a current is passing through the anode does not explain all observed facts, but they can be well explained by the theory developed for the positive and negative difference effects. (auth)—ALL. 15958

3.8.4, 3.4.8

Kinetics of Reaction of Steel with Hydrogen Sulfide-Hydrogen Mixtures. Andrew Dravnieks and Carl H. Samans. Standard Oil Company. J. Electrochem. Soc., **105,** No. 4, 183-191 (1958) April.

The kinetics of the reaction of steel with pure hydrogen sulfide at 250-500 C may be interpreted in terms of consecutive linear and parabolic rates. In hydrogen sulfide-hydrogen mixtures similar reactions exist, but the reaction rate decreases as the thermodynamic equilibrium line ($Fe + H_2S \rightleftharpoons FeS + H_2$) is approached, in a manner which is approximately proportional to the decrease in the thermodynamic driving force. Increase of pressure up to 20 atmospheres increases the reaction rate by a fractional power of the pressure. In the presence of traces of oxygen, the linear rate component is minimized. The observed kinetics can be explained in terms of three steps: adsorption, rate of formation of diffusing species, and diffusion. 16021

3.8.4, 3.5.9, 6.4.4

Oxidation of Magnesium by Air at Moderately High and High Temperatures. A. P. Belyaev. J. Applied Chem, USSR (Zhur. Priklad. Khim.), **30,** 1397-1400 (1957); Chem. Absts., **52,** No. 3, 1884 (1958) Feb. 10.

The rate of oxidation V of magnesium cylinders, 20 mm in diameter and 30 mm high, in air was determined at 200-265 C by the gain in weight. To prevent possible loss of scale the cylinders were exposed and weighed in porcelain crucibles. At 200 and 400 C $V = 0$ after an exposure of 8 hr. At 560° some oxidation occurred, $V = 0.07$ mg/sq cm/hr at 3-hr exposure. At 600 C the values of V after 1-, 1.5-, and 2-hr exposure were 2.1, 31.6 and 18.8 mg/sq cm/hr and burning started at 3-hr exposure. At 625 C V at 6, 8 and 10 min was 10, 30 and 33 mg/sq cm/hr, respectively, and began to burn after 15 min. These data supported Krilova's conclusion that the criterion of the ratio of volume of oxide to metal could not be used as an index of corrosion resistance.—ALL. 15515

3.8.4, 3.2.2, 4.6.2, 6.3.15

Kinetics of Scaling of Titanium in Steam Studied by a New Volumetric Method. (In German.) Karl Lönberg and Hans-Walter Schleicher. Z. Physik. Chem. (Neue Folge), **15,** Nos. 1-6, 223-224 (1958) April.

Measurement of consumed amount of steam and generated gas bubbles determines the course of corrosion.—BTR. 16074

3.8.4, 6.3.15, 3.5.9

Investigation on the Oxidation Mechanism of Titanium. (In English.) P. Kofstad, K. Hauffe and H. Kjöllesdal. Acta Chemica Scandinavica, **12,** No. 2, 239-266 (1958).

Four different rate laws were found in the indicated temperature intervals: logarithmic (<300 C); cubic (300-600 C), parabolic (600-850 C) and Parabolic + linear (>850 C).—BTR. 16072

3.8.4, 6.3.13

Oxidation of Tantalum at 50-300 C. D. A. Vermilyea. Gen. Elec. Research Lab. Acta Metallurgica, **6,** No. 3, 166-171 (1958) March.

Thickness of very thin tantalum oxide films can be estimated quickly and accurately from measurements of electrical capacitance of the films. Kinetics of growth of tantalum oxide films at temperatures below 350 C are consistent with equation developed by Cabrera and Mott. Rate-determining step for oxidation process is probably not at the metal-oxide interface. Oxide films formed up to 350 C are initially amorphous. Transformation to crystalline film is nearly complete after 1 day at

350 C, while only a small amount of crystallization occurs during oxidation for 4 days at 300 C. Oxygen dissolves into tantalum at 350 C rapidly enough to cause embrittlement after a few hours. Graphs, photomicrographs.—INCO. 15595

3.8.4, 6.3.9, 3.5.9

The Oxidation Rate of Molybdenum in Air. E. S. Bartlett and D. N. Williams. Battelle Memorial Inst. Trans. Met. Soc. AIME, **212,** 280-281 (1958) April.

In a recent program at Battelle, it became desirable to know more about the characteristic oxidation behavior of molybdenum under varying conditions of temperature and atmosphere. Using oxidation-test apparatus designed for dynamic, continuous recording of weight change during testing, values for the oxidation rate of molybdenum were obtained at temperatures from 1400 to 2150 F. In addition, the effect of air flow on oxidation rate was studied. The results of tests suggest that there is a maximum oxidation rate for molybdenum at a given temperature which is obtained when conditions are maintained such that the partial pressure of molybdenum trioxide in the atmosphere surrounding the specimen is at a low value.—NSA. 15941

4. CORROSIVE ENVIRONMENTS

4.6 Water and Steam

4.6.2, 5.4.5

Prevention of Corrosion in Steam and Condensate Systems. T. B. Fielden. Steam Engineer, **27,** 123-125 (1958) Jan.

Measures to combat steam condensate corrosion by use of filming amines. 7 references.—MR. 15618

4.6.12

New Trends in Treating Waters for Injection. J. W. Watkins. U. S. Bureau of Mines. World Oil, **146,** No. 1, 143, 145-150 (1958) Jan.

Discussion of recent developments in treating of brines and separation of oil from accompanying produced water. New methods for removing dissolved acidic gases from waters include controlled aeration, scrubbing with combustion gases, use of submerged burners and chlorination. Organic treating chemicals are finding extensive experimental application as biocides, corrosion inhibitors and wetting agents. New and improved chemical feeders and sedimentation tanks and ponds have been designed. Changes in filtration procedures are seen in development of corrosion-resistant element type filters and increased use of diatomaceous-earth filtration. Non-corrodible plastic and cement asbestos materials and corrosion resistant metallic alloys such as Monel are used extensively for many purposes. Automatic controls are utilized to a greater extent. 39 references, photos.—INCO. 15519

4.6.2, 7.6.4, 6.2.5, 6.2.4

Corrosion in High-Temperature Boilers. M. Werner. *Werkstoffe und Korrosion.* **8,** 587-596 (1957) Oct.

Review of German experience on behavior of materials in steam plant operating at 600 C after 40,000 hours' operation. Little corrosion was observed on water side since purity of steam can be readily ensured. Corrosion was only severe in presence of large amounts of impurities. Fire-side corrosion was not readily controlled, in view of deleterious influence of trace elements, such as sodium in ash. Supporting data are given on austenitic nickel-chromium, nickel-chromium-molybdenum and nickel-chromium-molybdenum-vanadium steels, low-alloy molybdenum and chromium-molybdenum steels. Some creep data and reference to dangers of stress-corrosion cracking and sulphide contamination are included.—INCO. 15147

4.7 Molten Metals and Fused Compounds

4.7

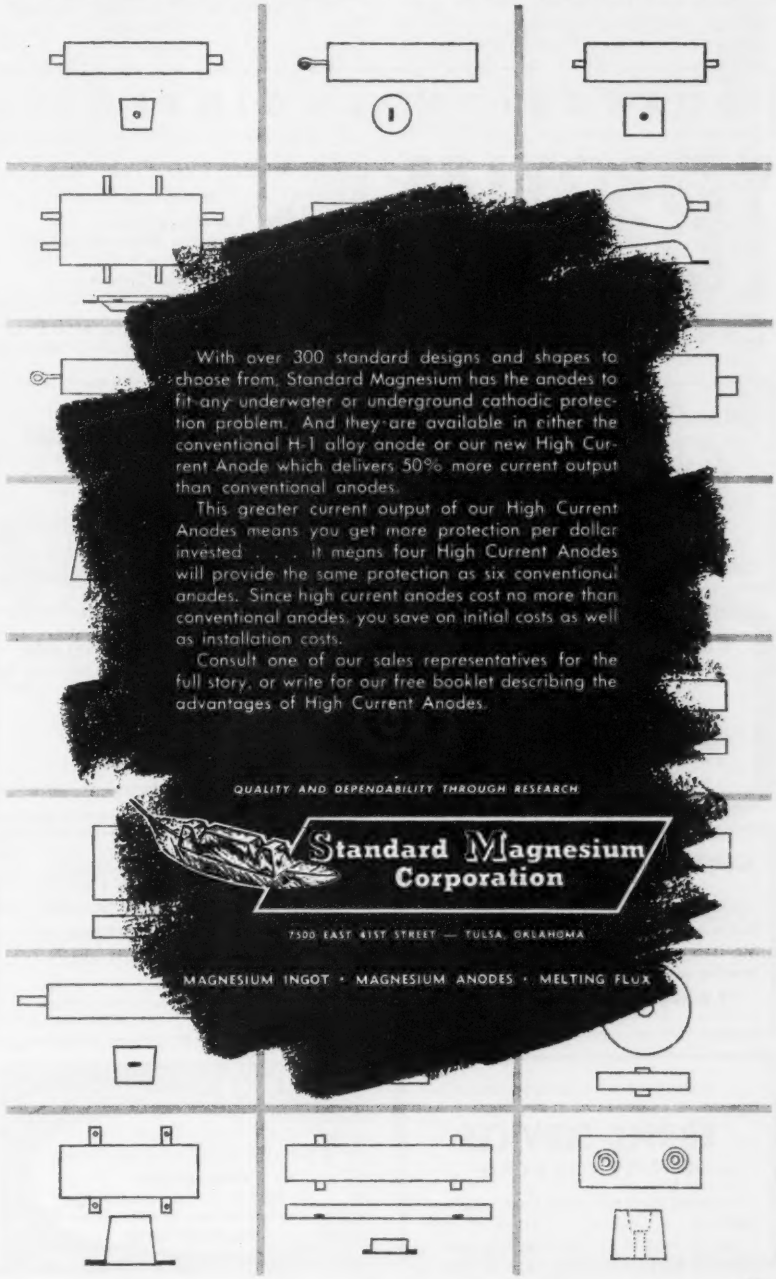
Corrosivity, Handling and Transfer of Molten Lithium. Olin Mathieson Chemical Corp., Niagara Falls, N. Y. and Syracuse Univ., N. Y. U. S. Atomic Energy Commission Pubn., Feb. 14, 1958, 118 pp. Contract AF33(600)-33920.

Problems of handling and corrosion during the electrolysis of lithium-chloride in a 1000 amp electrolytic cell and in the hydrogenation of lithium to lithium hydride are described. The corrosive effects of molten lithium on various materials of construction were investigated.—NSA. 15650

4.7

Corrosion Activity of a Nitrate-Nitrite Fusion. (In Russian.) E. I. Gurovich and G. P. Shtokman. *J. Applied Chem.,* USSR (Zhurnal Prikladnoi Khimii), **30,** No. 10, 1547-1552 (1957) Oct.

The effect of potassium nitrate (53%), sodium nitrite (40%) and sodium nitrate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(7%) on seven steels is tested. Selection of the best steel for construction of equipment and pipe lines for industries dealing with these fused salts. 4 figures, 14 references 15290

4.7

Static Liquid-Metal Corrosion. Interim Report Covering the Period July 1949-September 1952. ANTON DeS BRASUNAS. Oak Ridge National Lab. U. S. Atomic Energy Commission Pubn., ORNL-1647, September, 1952 (Declassified March 12, 1957), 99 pp. Available from Office of Technical Services, Washington, D. C.

Static corrosion tests have been made in molten sodium, lithium, lead, bismuth and several alloys containing uranium. Temperatures have been varied but the bulk of the data is concerned with tests made at 1000 C (1832 F). Sodium was found to be the least corrosive in static and dynamic tests, and almost all alloys tested have shown good corrosion resistance. Lithium, on the other hand, attacked many alloys quite severely, especially nickel and nickel-containing alloys. Thermal-gradient dynamic tests have also shown lithium to be much less attractive than sodium because of the susceptibility to mass transfer. Lead and bismuth were quite similar to lithium in many respects, both in static and dynamic corrosion tests and are compatible with very few metals at 1000 C (1832 F). (auth.)—NSA. 15015

4.7

The Reaction of Molten Metal with Water. Progress Rept. for May 1 through June 30, 1956. Aerojet General Corp. U. S. Atomic Energy Commission Pubn., AGC-AE-19 (Pt. I), July 23, 1956 (Declassified Jan. 15, 1957), 13 pp. Available from Office of Technical Services, Washington, D. C.

Experiments were initiated to determine the effect of sudden mixing of molten metal with water. Molten-metal drop tests and corrosion tests were carried out on zirconium, Zircaloy-2, and Zircaloy-B with 0.9% beryllium added. Oxide film thicknesses were determined and photomicrographs were made. Results are presented of molten-metal spray tests which were carried out on aluminum and zirconium.—NSA. 16135

4.7, 5.8.3

Zirconium and Titanium Inhibit Corrosion and Mass Transfer of Steels by Liquid Heavy Metals. O. F. Kammerer, J. R. Weeks, J. Sadofsky, W. E. Miller and D. H. Gurinsky. Brookhaven National Lab. Trans. Metallurgical Soc. AIME, **212**, No. 1, 20-25 (1958) Feb.

Zirconium and titanium inhibit mass transfer of steels by liquid bismuth, mercury and probably lead by reacting with surface of these steels to form adherent deposits of zirconium nitride, titanium nitride, or titanium nitride plus titanium carbide. These deposits are inert to

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liquid bismuth and lead-bismuth, and probably to mercury and lead. Data are presented which describe condition under which deposits form. Rate of formation is probably controlled by activity of nitrogen in steels. Rate of mass transfer in inhibited systems with large solubility gradient is controlled by rate of solution of steel into bismuth in hottest portion of circuit. In inhibited systems with low solubility gradient, rate of mass transfer is at least partially controlled by ability of inhibiting layer to promote or catalyze nucleation of precipitating iron or chromium in coldest portion of circuit. Graphs give data for pure iron, 5 chromium steel and 12 chromium steel. Tables, photomicrographs.—INCO. 15529

4.7, 6.3.15, 1.3

The Feasibility of Using Molten Metals for the Solution Heat Treatment of Titanium Sheet Alloys. D. J. Maykuth and E. S. Bartlett. Battelle Memorial Inst. Titanium Metallurgical Lab., Columbus, Ohio, December 19, 1957, 14 pp.

This memorandum summarizes a literature survey on the reactions of titanium with molten metals and the results of a brief laboratory program investigating the feasibility of using molten metals for the solution heat treatment of titanium sheet alloys. (auth.)—NSA. 15868

4.7, 6.6.6, 3.5.9

How Molten Aluminum Affects Plastic Refractories. H. A. McDonald, J. E. Dore and W. S. Peterson. J. Metals, **10,** No. 1, 35-37 (1958) Jan.

Three plastic refractory, alumina-silica compositions containing approximately 40, 60 and 85% aluminum oxide were tested. One lot of fire clay was used as the bond clay in all mixes. Test pieces 9 x 2½ x 2½ in. were rammed from each plastic composition. The samples of each were fired to 1600, 1800 and 2000 F and one 85% aluminum oxide piece was fired to 2400 F. All of the test pieces were exposed to 7075 aluminum alloy at 1400 F for 168 hr, this alloy being used because of its rapid attack on refractory materials. After completion of the holdings tests, penetration data were obtained by sectioning the test pieces with a diamond saw, parallel to the long dimension, and visually determining the extent of metal penetration into the refractory. It was found that the resistance of these materials to attack and penetration by molten aluminum is related to the uncalcined clay content and is particularly dependent upon the temperature to which the material is fired. When initially fired to temperatures below 1800 F plastic refractory materials show good resistance to attack and penetration by molten aluminum alloys. This resistance drops sharply when initial firing temperatures above 1800 F are used. The firing temperature sensivity of plastic alumina-silica refractories is related to the phase or crystalline changes which take place when clay, principally kaolinite, is heated. Amorphous metakaolin, formed when kaolinite loses its chemical water, is not readily wetted by molten aluminum. Thus, this bonding material provides a protective layer over the aggregate particles and minimizes attack and penetration. Metakaolin decomposes at temperatures above 1800 F and the resulting products, mullite and free silica, are susceptible to attack. Plastic refractories with a high clay content and low alumina content show the greatest resistance to penetration at firing temperatures below 1800 F. This resistance decreases with increasing alumina content and reduced clay content. This situation is reversed at firing temperatures above 1800 F where high alumina-low clay content plastics show the greatest resistance to penetration.—ALL. 15430

4.7, 8.4.5

Fused Salts as Thermonuclear Reactor Breeder Blankets. D. M. Gruen. Argonne National Lab. U. S. Atomic Energy Commission Pubn., ANL-5840, March 1958, 8 pp. Available from Office of Technical Services, Washington, D. C.

The use of fused lithium nitrate as a breeding material for thermonuclear reactors is proposed. Some of the properties of lithium nitrate such as thermal stability, radiation stability, neutron reaction products and corrosive effects are briefly considered. Further studies are suggested.—NSA. 15962

5. PREVENTIVE MEASURES

5.3 Metallic Coatings

5.3.2

Corrosion Behavior of Artificially Produced Hard Zinc Coats. (In German.) W. Dädeker. Metalloberfläche, **12,** 102-104 (1958) April.

Composition and some properties of zinc-iron alloyed coat; heat treatment. Corrosive effect of atmosphere on treated coat in laboratory and in natural conditions; optimal treatment temperature; limited application of method in practice.—MR. 15648

5.3.4

The Electroplating of Screws and Other Small Articles. 1. Quality Requirements and Preliminary Treatments. (In German.) W. Peters. *Draht,* **8,** No. 1, 7-13 (1957) Jan.

For optimum corrosion resistance, screws are electroplated with zinc or cadmium. Zinc is cheaper and withstands corrosion as well as cadmium in most environments, especially when it is chromate-treated. It can be bright plated to yield a pleasing surface, is more resistant to traces of chlorine, offers better galvanic protection and greater resistance to contact corrosion. Cadmium, on the other hand, is better for components likely to be soldered (e.g. electrical contacts), is more resistant to tarnishing and to corrosion indoors and by perspiration. Specifications for the thickness of electro-deposits are discussed and methods of cleaning, degreasing and 'activating' the screws before plating are described.—ZDA. 15003

5.3.4

Electroplating from the Pyrophosphate Bath. Pt. 2. Electrodeposition of Alloys. T. L. Rama Char. *Electroplating & Metal Finishing,* **10,** No. 12, 391-392, 408 (1957) Dec.

Summarizes work, both published and unpublished, that has been carried out in the Electrochemistry Laboratory of Indian Institute of Science on electrodeposition of alloys from pyrophosphate baths. Laboratory scale experiments indicate that pyrophosphate solutions may be successful for production plating of brass, alloys of tin with zinc, nickel, lead, and copper, alloys of nickel with copper, zinc, cobalt, manganese and tungsten and possibly also for nickel-iron, zinc-iron, tin-cobalt and alloys of rare metals like molybdenum. Tables list plating data for tin-nickel, tin-zinc, tin-lead, nickel-copper, tin-copper and zinc-copper.—INCO. 15037

5.3.2

Aluminum Coated Steel. W. E. McFee. Iron Age, **181,** No. 6, 95-97 (1958) Feb. 6.

Sheet steel coated on both sides with aluminum possesses surface characteristics of aluminum and physical and mechanical properties of steel. Materials resist atmospheric corrosion to serve a wide variety of outdoor prducts. Standard fabrication procedures for cold rolled steel must be modified.—MR. 16078

5.3.4

Mechanical Properties of Electrodeposited Copper as Depending on Electrolysis Conditions and Electrolyte Composition. (In Russian.) A. M. Ginberg and Yu. A. Klyachko. Metalloved. I Obrabotka Metal., No. 2, 35-37 (1958) Feb. Translation available from Henry Brutcher, Technical Translations, P. O. Box 157, Altadena, California.

Study of possibilities of controlling the tensile strength and elongation of electrolytic copper by varying the current density and introducing suitable surface-active substances into the bath. Experimental procedure and results. Minimum current density required for texturization of copper deposits in the presence of a surface-active substance. 2 figures.—HB. 15636

5.3.4

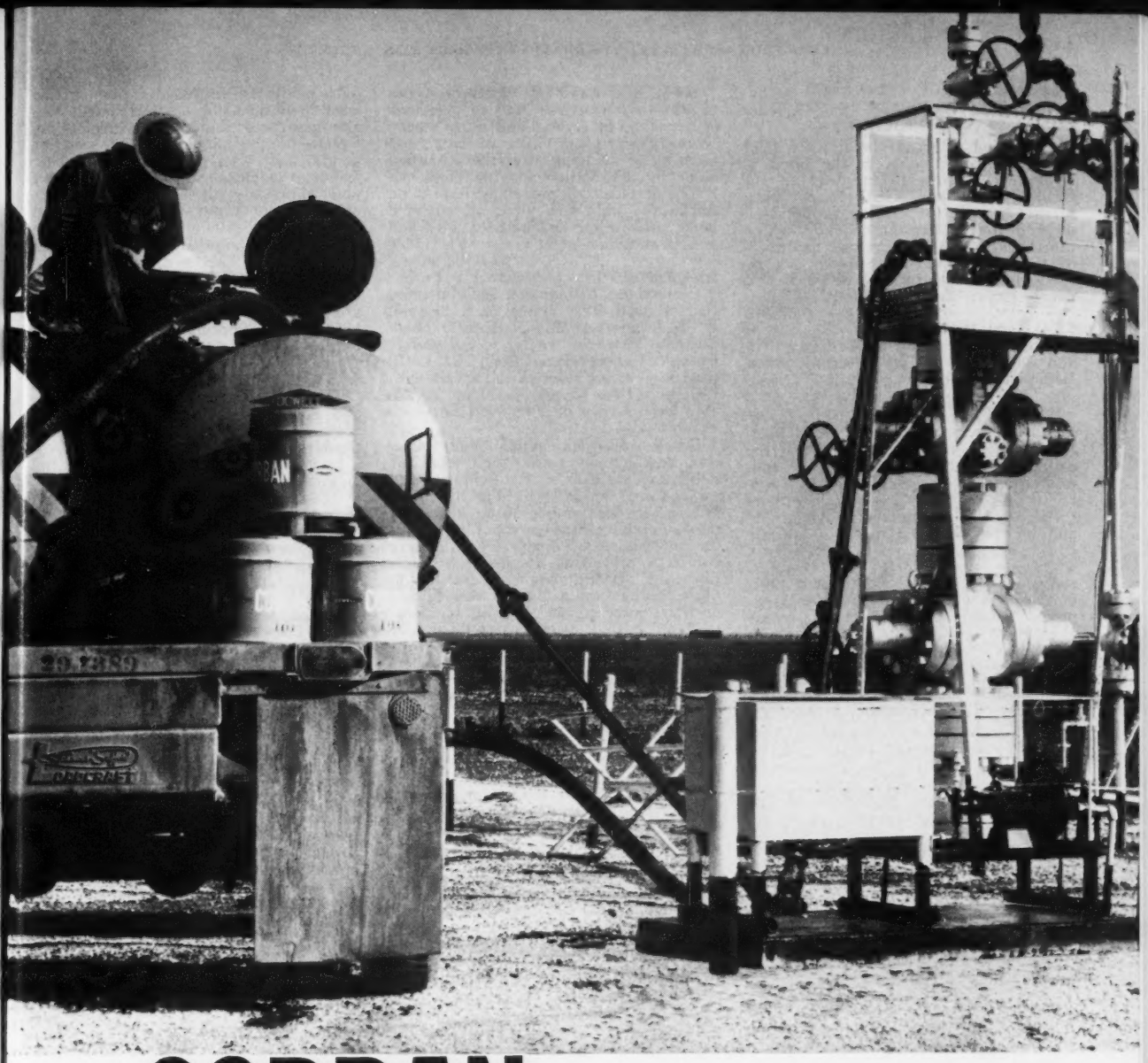
Coatings of Nickel-Aluminum Alloys Prepared by Electrolysis. Period Covered: January 1957 to January 1958. Dwight E. Couch and Jean H. Connor. National Bureau of Standards. U. S. Wright Air Development Center, Technical Report No. 58-5 (Pt. I), Feb. 15, 1958, 37 pp.

The electrodeposition of nickel-aluminum alloy coatings for the protection of Thermold J Alloy steel was investigated because of certain properties of the alloy, namely, extreme hardness and oxidation resistance. The alloy was formed by plating aluminum upon a layer of nickel. When the fused salt baths operated above the melting point of aluminum, the alloy was formed directly. When the aluminum was deposited at lower temperatures, the samples were later heated to form the alloy by diffusion. Oxidation data and salt spray corrosion resistance of the alloy-plated steel are given. Optimum protection of the steel was afforded by the alloy formed at 700 C, consisting of 2 mils of nickel and 0.2 mil of aluminum. (auth.)—NSA. 15957

5.3.4

A Comparative Study of Non-Ferrous Coatings on Steel. A. N. Kapoor, A. B. Chatterjea and B. R. Nijhawan. Symposium on Non-Ferrous Metal Industry in India 1954, **1957,** 256-265.

The galvanizing, tinning and aluminizing processes are outlined. The microstructures obtained in the coatings formed by the processes are compared together with their corrosion-resistance. Aluminized steel as an alternative to zinc and tin on steel is suggested in view of the large bauxite reserves in India and the need to import large tontages of tin and zinc. 26 references. —MA. 15812



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5.3.4
Vinyl-Clad Steel. Ind. Eng. Chem., **49,** 93A-94A (1957) July.

A method of vinyl cladding of steel was developed wherein the bond is strong enough to withstand drawing and forming operations. The process makes possible a continuous mechanized method for coating metal. The major drawback is the temperature limitation of the vinyl film. Flat pieces can be used at temperatures up to 250 F and deeply drawn parts up to 160 F.—PDA. 15818

5.3.4
A Corrosion Study of Cadmium-Tin and Zinc-Tin Alloy Electrodeposits. Bennie Cohen. Plating, **44,** No. 9, 963-968 (1957).

Tests are described on electrodeposited 73:27 cadmium-tin and 62:38 zinc-tin alloys on steel which show that the cadmium-tin alloy is superior to cadmium under salt-spray test, slightly better in high-humidity conditions and equal in galvanic effect upon aluminum and magnesium alloy in high-humidity conditions. In these tests the zinc-tin coatings were inferior to both cadimum and cadmium-tin alloy. In additional tests on the cadmium-tin alloy it proved superior to cadmium in corrosion-resistance to jet fuels containing mercaptans, to high-temperature synthetic oils, and in the presence of the vapors from aqueous solutions of acetic acid. It was easily solderable by methods used for cadmium, and at low temperature, the tin-rich phase was not converted from white to grey tin.—MA. 15782

5.3.4, 3.4.7
Investigation into Variations of pH Value in a Non-Electrolytic Nickel Plating Solution. L. Bosdorf and K. Muller. Metalloberfläche, **12,** 150-151 (1958) May.

Study of variation of pH in non-electrolytic nickel plating solution (containing 30 g of nickel chloride, 2.5 g cobalt acetate and 10 g of sodium hypophosphite per liter). Investigation included effect of sodium glycolate on pH. A 34.5 micron thick nickel deposit formed from above solution during two hours, pH falling from 3.5 to 3.89.—INCO. 16050

5.3.4, 6.4.2
Firm Adherent Plating for Aluminum. J. C. Withers and P. E. Ritt. Melpar, Inc. Metal Finishing, **56,** No. 1, 53-54, 57 (1958) January.

Presents details of new method of obtaining electrodeposits on aluminum with excellent adhesion. Method consists of etching aluminum article in hydrofluoric acid solution, etching again in hydrochloric acid and manganese sulfate, plating from nickel sulfate bath, heat treating at furnace temperature of 800-1050 F for 5 minutes, and subsequently plating with any desired metal. Heat treating the nickel-clad aluminum results in formation of islands of aluminum-nickel alloy which provide suitable surface for further plating. Photomicrographs compare nickel-flashed aluminum before and after heat treatment. Composition of nickel bath is given.—INCO. 15487

5.3.4, 6.2.3
The Effect of Gas- and Salt-Bath Nitriding on the Properties of Structural Steels. Pt. I. H. Wiegand and M. Koch. Metalloberfläche, **12,** 69-74 (1958) March.

Short description of different types of nitriding processes and mechanisms of nitriding in carbon and alloy steels. Note is given on effect of nickel on ductility of nitriding steels, when added alone or with other alloying elements. Study of effect of process variables on thickness and structure of nitrided layer, and fatigue strength of a carbon steel was made.—INCO. 15480

5.3.4, 6.3.9, 3.5.9
Protective Coatings for Molybdenum. C. F. Powell, R. P. Jones, A. C. Secrest, E. M. Sherwood and I. E. Campbell. Battelle Memorial Inst. U. S. Atomic Energy Commission Pubn., BMI-810 (Del.), Feb. 17, 1953 (Declassified with Deletions Feb. 13, 1957), 30 pp. Available from Office of Technical Services, Washington, D. C.

Fifteen-mil-thick molybdenum sheet was satisfactorily protected from air oxidation at 2200 F by being coated (siliconized) at 2550 F in a hydrogen atmosphere containing 10 to 27 vol.% of silicon tetrachloride vapor. Coatings varying between 3.8 and 6.4 mils gave complete protection at the test conditions for periods ranging from 138 to 616 hr. The vapor-deposited silicide coatings were brittle at room temperature and coated specimens could not be bent cold. The addition of chromium or phosphorus to the vapor-deposited coatings did not improve the ductility or oxidation resistance of the coatings. Painted-and-sintered coatings of nickel-chromium-silicon powder mixtures were also highly protective to molybdenum in air at 2200 F, but were not as reliable as the vapor-deposited coatings. However, protection for periods as long as 190 hr was obtained with 3-mil-thick coatings on a significantly large number of specimens. These coatings had advantages over vapor-deposited coatings in that they were ductile at room temperature, and could be more easily applied to large, irregularly shaped objects. (auth.)—NSA. 15903

5.3.4, 8.8.3
Technical Comments on Some Treatments of Machine Parts by Hard-Chromium Plating. P. Morisset. Metaux-Corrosion-Industries, **32,** No. 381, 208-213 (1957).

Applications discussed include the plating of engine parts, brake drums, textile-machinery parts, hydraulic jacks, water turbines, machine-tool components, cinema-industry plates and cylinders and musical instruments. Hard-chromium plating of electropolished parts, aluminum and aluminum alloys, and for protection against sea-water corrosion is also considered.—MA. 15866

5.4 Non-Metallic Coatings and Paints

5.4.5
Calcium Plumbate in Priming Paints. N. J. Read. J. Oil Col. Chem. Assoc., **41,** No. 5, 352-362 (1958).

The properties of calcium plumbate as an anticorrosive pigment, first reported in 1950, are shortly recapitulated. Seven years' commercial use of calcium plumbate primers confirms conclusions drawn from the initial laboratory experiments and establishes the pigment as a prominent member of the limited number of anticorrosive pigments for the protection of iron and steel. The versatility of calcium plumbate paints is discussed in connection with their behavior on galvanized iron, aluminum and timber. The chemical behavior of the pigment in linseed oil paints has been examined. Although lead and calcium soaps exist in the dry paint film, only calcium compounds are extracted by water, yielding solutions having pH 8-9. The effects of the lead and calcium compounds on film formation and the influence of the water-soluble calcium compounds on the behavior of calcium plumbate oil paints are discussed. The use of calcium plumbate in quick-drying and stoving dipping paints is described.—RPI. 16115

5.4.5
The Use of Mixtures of Zinc Oxide and Potassium Chromate in Wash Primers. H. F. Clay and V. Watson. Electroplating, **11,** No. 3, 89 (1958) March.

In general, zinc tetroxychromate, the pigment originally used in wash primers of the two-pack types, was found to be slightly superior to all other pigments tested with the exception of a mixture of zinc oxide and zinc potassium chromate. 2 figures.—ZDA. 15614

5.4.5
Protective Coatings Based on Bitumen. J. Stanford. Corrosion Prevention and Control, **5,** No. 3, 62-64 (1958) March.

Considers nature of bitumen and how far bitumen meets requirements of an ideal protective coating. Versatility of bitumens in present-day practice is demonstrated. Most satisfactory colored bituminous coating is that obtained by addition of metallic aluminum. Apart from pleasant appearance of metal surface formed when aluminum settles out, it also protects underlying bitumen from sunlight and prevents oxidation. Proper surface preparation is indicated.—INCO. 15494

5.4.5
Metal Pretreatment. C. D. Coppard. Paint Tech., **21,** No. 234, 89-90 (1957).

Three metallic lead primers and one ship's bottom primer were applied to rusty steel plate which had been wire brushed or treated with a phosphoric acid-based solution. Panels were tested in humidity and salt spray cabinets. The results clearly showed the benefit of the phosphoric acid treatment.—RPI. 15756

5.4.5
Vinyl-Resin-Based Paint. E. S. Lower and S. Cressey. Corrosion Technology, **4,** No. 6, 189-191 (1957).

Vinyl resin coating systems are no novelty to those concerned with the protection of ship bottoms against fouling and corrosion, because it was for this purpose that they were originally developed during the 1939-45 war. However, this article is intended to draw attention to the use of such systems for a rather different purpose, viz., the protection of steelwork against highly corrosive conditions. This is a logical step resulting from observation of the outstanding protection given by vinyl resin coatings against very corrosive marine conditions.—RPI. 15822

5.4.5
Looking into Paint. Pt. II. Typical Surfaces. J. Lawrance. Painting and Decorating, **77,** Pt. 917, 34, 36 (1957).

A consideration of the characterization of wood, plaster and metal in terms of porosity, rigidity and corrosive influences.—RPI. 15984

5.4.5

Epoxide Resins Versus Corrosion. L. M. Barakan and G. L. E. Wild. Corrosion Technology, **5,** No. 5, 137-140 (1958) May.

Covers essential nature of various types of epoxide finishes, their usefulness and respective merits. Under consideration are: epoxide resin-containing coatings which dry at ordinary temperatures by oxygen absorption; cold-setting or catalyzed epoxide-based coatings which harden at ordinary temperatures through chemical reaction; stoving epoxide coatings which can be stove-hardened at 250-300 F; and stoving epoxide coatings which are hardened at 350-400 F. Incorporation of corrosion inhibiting pigments (zinc, red lead, zinc chromate, etc.) in epoxide media is desirable.—INCO. 16048

5.4.5

Looking into Paint. Pt. I. Surface Factors. J. Lawrance. Painting & Decorating, **77,** Pt. 916, 42, 44 (1957).

The nature of the surface of the substrate is an important factor in painting. A rough surface often gives better adhesion and durability than a smooth; dimensional changes due to changes of temperature and humidity must be allowed for. Brushing is better than spraying on porous surfaces.—RPI. 15983

5.4.5

Anti-Rust Additives in Primer Paints. N. Estrada. Official Digest, **29,** No. 394, 1077-1085 (1957) Nov.

A research project is described in which additions of organic compounds were made to conventional primers and the corrosion resistance of the resultant paints was tested in a salt spray cabinet. Two of the primers contained considerable proportions of zinc chromate, while a third contained zinc chromate and zinc oxide. The results showed that two of the thirteen additives were definitely beneficial to those primers which already had corrosion resistant properties, i.e. to those containing zinc chromate and/or red lead. 2 figures.—ZDA. 15765

5.4.5

Plastic-Based Strippable Coatings. E. S. Lower and S. Cressey. Corrosion Technology, **4,** No. 3, 83-85 (1957).

The growth of plastics as corrosion-resistant coatings is a relatively recent one. Until the Second World War their application in this field had hardly been initiated, but once the demand was created the manufacturers responded wholeheartedly and a large number of coatings, with different characteristics and applications, were developed. This article is concerned with some of these developments.—RPI. 15830

5.4.5

Adhesion of Wash Primers. B. Wäser. Adhäsion, **2,** No. 1, 8-10 (1958).

The effect on adhesion to (measured in the Ericksen apparatus) and protection against corrosion of iron surfaces, of modification of a standard wash primer with phenyl ethyl sulfonate, polyoxyethylene sorbitol laurate and polyvinyl chloride is reported. Adhesion and corrosion resistance were a maximum at 4-5% additions.—RPI. 16004

5.4.5

Protection of Metals by Polyvinyl Butyral—Phosphoric Acid-Chromium Derivative Complexes. P. Duval. Corrosion et Anti-corrosion, **5,** No. 6, 189-193 (1957).

Advantages of wash primers and details of their composition and formulation are presented and discussed. The primers are applied on cleaned, degreased surfaces by brushing, spraying, or dipping and they dry very rapidly to give a base to which most paints adhere well. Special reference is made to protective-painting application.—MA. 15562

5.4.5, 2.2.5, 2.3.2, 6.4.2, 4.2.5

Studies Concerning the Behavior of Lacquer Coatings on Aluminium in a Marine Atmosphere. (In Italian.) G. Luft, Alluminio, **27,** No. 3, 107-118 (1958) March.

To determine the protective value, on aluminum, of various paint systems, painted test specimens, prepared from aluminum-4% copper-magnesium, and aluminum 3½ magnesium alloys, were subjected to corrosion by salt spray and marine atmospheres. Tests were carried out in natural surroundings and also by using laboratory methods. (Weatherometer). Experimental results indicate that best corrosion protection is provided by the application of an alodine treatment or a wash primer, followed by a lacquer coating composed of a PVC vehicle, zinc chromates and silicates.—ALL. 15646

5.4.5, 3.3.2, 8.9.5

Why Shipbottom Paints Fail. S. Miller. University of Miami. Sea Frontiers, **4,** No. 1, 13-22 (1958) Feb.

Fouling can be prevented from attaching to any type of hull only by application of an antifouling paint, and each type of hull requires its own special treatment in application. Various causes of failures of antifouling paints are discussed. Photos.—INCO. 15470

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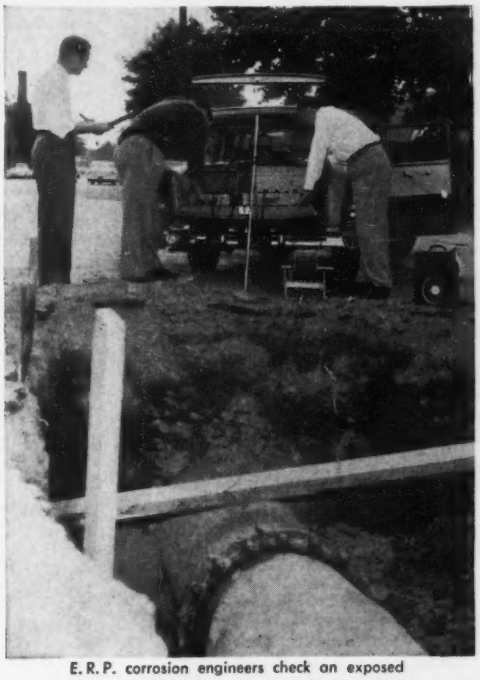

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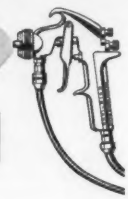

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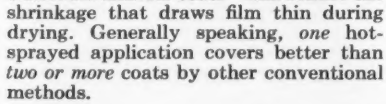

Demonstration on two glass panels proves how heavier film-build — thus, greater protection — is possible with hot spray. Left panel shows paint applied to its maximum film thickness by cold-spray method, and right panel shows how much heavier the same paint can be applied by DeVilbiss Hot Spray Method.

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5.4.5, 3.7.3

Painting Weldments. R. Clement. Prat Soud., **11,** 29-33 (1957) Feb.; J. Iron and Steel Inst., **188,** Pt. 2, 192 (1958).

Discusses the deterioration of paints over weldments. The principal causes of corrosion and the action of rust on oil paints are described. Means of overcoming paint deterioration are reviewed. —RPI. 15760

5.4.5, 5.4.8

New Anti-Corrosive Pigment. R. P. Bates. Am. Paint J., **42,** No. 30, 34, 38 (1958).

The qualities and uses of basic lead silicochromate pigment, claimed to consist of an inert core surrounded by an active surface, are described. The analysis given is basic lead chromate 29.1, lead silicate 25.5 and silica 45.4%. Particle size is stated to be 5 μ and coat thickness 2 μ. It has low tinting strength. —RPI. 16046

5.4.6, 5.4.8

The Value of Specifications in Decorative Nickel Plating. H. C. Castell. Mond. Metal Finishing J., **4,** No. 40, 119-121 (1958) April.

Factors contributing directly to early breakdown of nickel plated articles are reviewed, namely, ready-made pores and discontinuities arising from co-deposition of insoluble particles suspended in solution, gas-bubble pitting, cracks in deposit and penetration of deposit by corrosion. First two factors have been practically eliminated in present-day plating techniques. With judicious use of anti-stress agents and regular carbon purification of solution, danger of cracks has been lessened. With reference to corrosion, atmospheric exposure tests by ASTM showed that thickness of nickel layer, applied directly to steel or to copper undercoat, is factor which principally determines length of life of plating. Therefore, when plater is asked to provide decorative nickel-chromium finish, he must consider environment in which article is to be used and thickness of deposit necessary to ensure freedom from visible deterioration for long period of time. Specification is proposed in which particular attention has been given to type of service and minimum thickness necessary. (table.) —INCO. 15937

5.4.5, 6.4.1

Painting of Non-Ferrous Metals. (In German.) K. A. van Oeteren-Panhäuser. Metalloberfläche, **12,** No. 2, 53-56 (1958) Feb.

Among the non-ferrous metals surveyed from the point of view of corrosion-protection by painting, aluminum and magnesium are also included, although, under normal conditions, they do not need protective coatings. Certain aluminum alloys, however, are less resistant to corrosion than the pure metal itself; acids, alkalis, salts and especially chlorides are detrimental to aluminum, and so is sea water to magnesium. In such applications protective coatings of the metal is necessary. Practical recommendations are given concerning pretreatment, priming, selection and application of the paint.—ALL. 15483

5.4.5, 7.7

Epoxide Coatings for Large Diameter Electrical Conductors. V. and M. Hanzlik and J. Mleziva. Chem. Prumysl, **7,** No. 12, 673-678 (1957).

The authors have studied the influence of acid hardening and plasticizing components on the technological properties of hardened films of a three-component epoxide coating. The results are applied to formulate an electrical insulating coating.—RPI. 15854

5.4.5, 8.4.3

How Epoxies Can Simplify Maintenance. E. R. Scogin and H. W. Howard. Shell Chemical Corp. Petroleum Refiner, **37,** No. 1, 152-154 (1958) Jan.

Epoxy resins, properly formulated into coatings and applied, are simplifying corrosion maintenance problems and cutting the annual dollar loss due to atmospheric and chemical corrosion in oil refineries and petro-chemical plants. Surface coatings based on these resins are suited for this job because of their versatility and combination of properties they exhibit; excellent adhesion, flexibility, toughness and chemical resistance, thus combining in 1 resin the outstanding characteristics of many other types of resins. Different types of epoxies are described. Photos.—INCO. 15501

5.4.5, 8.9.5

Preservative Coatings for Ships in Service. L. S. Birnbaum. Bureau of Ships. Bureau of Ships J., **6,** No. 10, 2-6, 12 (1958) Feb.

Describes details involved in selection of Navy paints and furnishes information regarding some paints that are specified, or are under consideration, for standard Navy use. Surface preparation and pretreatment techniques are reviewed. Types of primers used are discussed. Paints are specified for each of the following areas: underwater hull from keel to lower boot topping limit (hot plastic, cold plastic and vinyl). waterline from lower to upper boot topping limit (black cold plastic and vinyl), appendages such as struts and rudders (thiokol), exterior decks (mastic and synthetic rubber), tanks, bilges and voids, and finally, aviation fuel tanks (seran).—INCO. 15451

5.8 Inhibitors and Passivators

5.8.2, 8.9.3

Cathodic Protection of an Old Uncoated Pipeline. D. P. Price and R. M. Wainwright. Gas, **34,** No. 3, 119-125 (1958) March.

Case history of application of "hot-spot" coating and cathodic protection to an old predominantly uncoated pipeline. Application followed situation of extensive leak history soon after gas pipeline was constructed, and resulted in greatly reduced maintenance costs. Although required protective currents are high, preservation of pipeline which is becoming increasingly more expensive to replace, more than justified cost of cathodic protection. History of the pipeline, soil conditions along the line, leak analysis, cathodic protection and pipe replacement program and economics of cathodic protection vs. replacement are covered.—INCO. 15639

5.8.2

Vapour-Phase Corrosion Inhibitors. S. Rowden. Corrosion Technology, **5,** No. 4, 117-121, 126 (1958) April.

Describes development of vapor phase inhibitors since 1945. Most important class of chemical agents used function by changing the electrochemical processes occuring at the metallic surfaces, e.g. chromate, nitrites and phosphates. Following are discussed as important characteristics of vapor phase inhibitors:

stability, water solubility, sufficient volatility to maintain and uphold adequate inhibiting concentration and acceptable diffusion characteristics in gaseous state. The three main types of organic substances which fulfill these requirements are amino and other nitrites, esters of carboxyl acid, and amine carbonates. Factors affecting use of vapor phase inhibitors are: acidic environments which convert amines into non-inhibiting nitrosoamines, instability at high temperature, time lapse before a protective atmosphere is produced, and effect on non-metals. Action of vapor phase inhibitors on different metals and industrial applications (packing and storage) are discussed.—INCO. 16001

5.8.2, 4.5.2, 6.2.2

On the Inhibition of the Corrosion of Buried Iron. T. Markovic, D. Zagar and N. Plavsic. Werkstoffe u. Korrosion, **9,** No. 2, 73-75 (1958) Feb.

Report on some laboratory experiments on inhibition of corrosion of buried iron. Results are explained in terms of influence of the physical properties of the soil upon the suppression of corrosion by sodium chromate, sodium nitrite and sodium benzoate inhibitors. Inhibitors are recommended for bare iron pipes in moist soil.—INCO. 15508

5.8.2, 4.3.3, 6.6.6

Inhibition of Alkaline Attack on Soda-Lime Glass. G. A. Hudson and F. R. Bacon. Am. Ceram. Soc. Bull., **37,** No. 4, 185-188 (1958) April.

Small quantities of a number of compounds, including zinc chloride, were added to 3% solutions of sodium hydroxide in order to determine the extent to which they reduced the attack of the solution on soda lime glass. Beryllium sulfate was found to reduce attack by 97% and zinc chloride by 90%. 5 tables. —ZDA. 15928

5.8.3

Electrocapillary Action of Corrosion Inhibitors. H. C. Gatos. du Pont. Nature, **181,** No. 4615, 1060-1061 (1958) April 12.

Tests are described which show that the order of relative inhibiting effectiveness of a number of amine inhibitors is the same as that of their effectiveness in suppressing certain polarographic maxima. These amines were most effective in suppressing maxima corresponding to negative branch of electrocapillary curve at which mercury is negatively charged with respect to solution. It was thus demonstrated that significant similarities exist between processes involved in polarographic maxima and their suppression, on one hand and in metallic corrosion and its inhibition on the other. Graph.—INCO. 15685

5.8.3, 3.8.4, 6.2.2, 4.3.2

Influence of Inhibitors on Ratio Between Adsorbed Hydrogen and Released Hydrogen in Corrosion of Iron in an Acid Medium. (In French.) Giampaolo Bolognesi and Liliana Felloni. Corrosion et Anticorrosion, **5,** 210-215 (1957) July-August.

Action of three typical inhibitors (OTT, ISOT, PTOL) on speed of dissolution of Armco iron in hydrochloric acid was studied by gasometric measurement of released hydrogen and colorimetric measurement of iron in solution. Since quantity of adsorbed hydrogen was quite large, measurements of volume of released hydrogen could not provide a precise evaluation of inhibiting power, particularly when readings were made after a brief delay. Choice of an inhibitor should depend largely on quantity of adsorbed oxygen. 19 references.—MR. 15715

5.8.3, 4.3.2, 6.2.2

Corrosion Inhibition by Organic Amines. H. Kaesche and N. Hackerman. University of Texas. J. Electrochem. Soc., **105,** No. 4, 191-198 (1958) April.

Corrosion of pure iron in 1 normal hydrochloric acid is discussed in terms of theory of mixed potentials and theory is applied to inhibition by organic compounds. Corrosion rates with and without inhibition by aniline, several aniline derivatives and alkylamines were determined by cathodic polarization measurements as well as colorimetric analysis of solution. All compounds show maximum inhibitor efficiency at concentration of approximately 0.1 mole/1, all are cathodic as well as anodic inhibitors and in most cases they are predominantly anodic inhibitors. Interpretation of data on cathodic inhibition is suggested on basis of assumption of uniform metal surface and uniform adsorption. Tables, graphs.—INCO. 15687

5.8.4

Corrosion Preventive Additives. Armour Research Foundation for Wright Air Development Center, U. S. Air Force, March 1952, Released June 17, 1958, 120 pp. Available from Office of Technical Services, U. S. Dept. of Commerce, Washington 25, D. C. (Order PB 131459).

Final report of a year (1951-1952) of Air Force-sponsored research aimed at development of new corrosion inhibitors to supplement or replace petroleum sulfonates as lubricant additives.

A study was made of petroleum sulfonates to determine the nature of the compounds showing corrosion inhibition. Separation of a commercial sodium petroleum sulfonate and fractionation of the sulfonates showed the sulfonates to be alkyl benzene derivatives with the alkyl group in the para position to the sulfonic acid group.

A large number of commercially available organic compounds were evaluated by the Static Water Drop test and a galvanic couple system. Several good inhibitors were found, and general information was obtained concerning the type of organic compounds which will inhibit galvanic corrosion.

The research produced many synthetic organic compounds showing corrosion inhibiting properties. Among these were glyoxalidines, alkyl aryl sodium sulfonates, amine salts of 2-ethylhexoic, oleic, nicotinic, pelargonic, linoleic, and dodecylbenzenesulfonic acids.—OTS. 15567

5.8.4, 4.3.2

Elemento-Organic Compounds of the Fifth Group of Mendeleev's Periodic System as Inhibitors of Acid Corrosion. S. A. Balezin and M. A. Ignatyeva. J. Applied Chem., USSR (Zhur. Priklad. Khim.), **29,** No. 11, 1777-1784 (1957). Translation by Consultants Bureau, Inc. (In Russian.)

Effects of organic compounds containing phosphorus, arsenic, antimony and bismuth on solution rate of steel in acids in relation to the concentrations of acid and inhibition. 6 references.—MR. 16140

Spraying tips for FIGHTING CORROSION

How to eliminate "thin" edges. Attaining adequate film-build on sharp edges, ridges, corners and projections is a problem with any protective-coating job.

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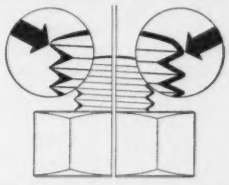

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5.8.4, 6.2.2

Organic Corrosion Inhibitors for Iron. ROBERT JENNY. *Ind. Finishing,* **9,** 872 + 4 pp. (1957) October.

Studies with liquid and insoluble mercaptans of different structures—BTR. 15332

5.9 Surface Treatment

5.9.1

Surface Preparation. PT. VI. J. SNELLING. *The Decorator,* **56,** No. 662, 53-54 (1957).

A discussion of the problems of rust-removal from iron and steel, with notes on the use of phosphating and on rust-inhibitive primers.—RPI. 15065

5.9.2

Pickling, Descaling and Derusting of Steels. W. Bullough. Metallurgical Reviews, **2,** No. 8, 391-406 (1957).

Comprehensive review covers theory of pickling, pickling of mild steel, electrolytic pickling, gas pickling, sodium hydride pickling, acid pickling of stainless steels, disposal of spent liquor and other methods of descaling such as blast cleaning and flame descaling. 51 references.—INCO. 15518

5.9.2, 6.2.5

Electrolytic Polishing of Stainless Steel. P. F. McKinney. Chem. and Process Eng., **39,** No. 2, 59-60 (1958) Feb.

Describes an electrolytic method of polishing steels, Electropol process, which has wide application in chemical and allied industries. Method produces a new type of surface finish which not only gives increased corrosion resistance and cleanliness, but also reduces costs and gives greater freedom at design stage. Photos.—INCO. 15444

5.9.2, 6.3.15, 3.2.3

Descaling and Cleaning of Titanium and Titanium Alloys. D. W. Stough, H. S. George, E. B. Friedl, W. K. Boyd and F. W. Fink. Titanium Metallurgical Lab., Battelle Memorial Inst. for Office of Assistant Secretary of Defense (R&D), March 28, 1958, 64 pp. Available from Office of Technical Services, U. S. Dept. of Commerce, Washington, D. C. (Order PB 121640).

A survey of the present commercial practices for descaling titanium and titanium alloys is presented. Scales formed on titanium and titanium alloys that have been heated in air at temperatures up to about 1100 F are generally removed by acid pickle baths. More drastic treatment is usually required when the metal is heated in air to temperatures in excess of 1100 F. Titanium with scales formed above 1100 F requires treatment in a molten-salt bath, which may utilize an electric current, followed by pickling in a suitable acid solution. Proprietary descaling methods are discussed to the extent that such information is available. Other methods currently in use to clean the surface include sand, grit, and vapor blasting, and a softening treatment in aqueous caustic, similar in sequence to the molten-salt bath. Descaling practices at the major producers of mill products, such as titanium billet, bars, plate and especially sheet, have been discussed in detail. The data are discussed generally, conclusions given and recommendations made for further research. (auth).—NSA. 15977

5.9.3, 3.5.8

The Effect of Different Surface Treatments on the Fatigue Strength of Drill Steel. T. W. Wlodek. Can. Mining and Met. Bull., **51,** No. 550, 89-98; disc. 98-101 (1958) Feb.

Relative merits of shot peening, induction surface hardening, spiral-rolling and combination of these surface treatments are evaluated on basis of their capacity to increase fatigue strength of drill steel. Sulfur-nitrogen relations for plain carbon steel and nickel-chromium-molybdenum drill steels in as-rolled condition are given. Magnitude of surface compression stresses and their effect on fatigue strength are discussed. Tables, graphs. 26 references.—INCO. 15602

5.9.4

Ferric Phosphate Precipitation in Phosphating Baths. S. L. Eisler & P. G. Chamberlain. Industrial Finishing, **9,** No. 113, 939-942 (1957).

Studies, using an isotope technique, were made on the solubility of ferric phosphate in different phosphating systems in order to reduce ferric phosphate precipitation in the sludge. It was concluded that considerable economics could be made by holding the nitrate content of the bath at an optimum level of 1-2.5%.—RPI. 15794

5.9.4

Thermodiffusion Plating of Metals. (In Russian.) N. A. IZGARYSHEV. *Trudy Komissii po Bor'be s Korroziei Metal.* Akad Nauk SSSR, **1956,** No. 2, 149-154; *Chem. Absts.,* **51,** No. 9, 6484 (1951).

In the thermodiffusion method of plating, a chloride of the plating metal is produced at high temperature; the gaseous chloride is made to diffuse into ing were investigated with different produces surface layers composed of a metallic compound, increasing the resistance of the original metal. The technique and results of this method of plating were investigated with different metals. Chromium was plated by using chromic chloride in a chlorine atmosphere. The optimum temperature range was 850-1000 C. The average concentration of chromium in the layer penetrating 0.2-mm in depth of iron was 15%, but the surface layer was much richer. It consisted of a solid solution of iron and chromium with traces of carbide in the case of high-carbon steel and was perfectly corrosion-resistant in air saturated with water vapour carrying 1% hydrogen sulfide or sulfur dioxide. The plated metal withstood heat-treatment and even rolling. Aluminum plating was done at 1000 C with aluminum chloride gas. The layer obtained was resistant to high temperature. Of special interest were the highly resistant layers of a mixture of aluminum + chromium. Also chromium + silver layers 0.183 mm thick were obtained; the protection against 3% sodium chloride solution was perfect. Molybdenum + tungsten and tin + copper plating was also successfully achieved.—MA. 14979

5.9.4, 3.5.9, 6.4.2

Effect of Temperature of Electrolyte on the Thickness of the Oxide Film and the Duration of Anodization of Aluminum and Aluminum Alloys. (In Russian.) I. M. Plotnikov. J. Applied Chem., USSR (Zhur. Priklad. Khim.), **30,** 1321-1325 (1957); Chem. Absts, **52,** No. 4, 2607 (1958) Feb. 25.

Aluminum plates, 120 x 50 x 1 mm., were anodized in sulfuric acid, 192 g/l, at 15-30 C with current density 1-3 amp/dm². The weight of the plate increased with the duration t of anodization and passed through a rounded maximum at t_m and then decreased. The best films were obtained up to t_m which also corresponded with the maximum thickness of the film. t_m decreased as the temperature increased. The maximum thickness at t_m increased with the temperature. To obtain the best films of the same thickness t should be decreased and the current density increased.—ALL. 15897

5.9.4, 6.4.2

Role of Chromate Treatments in the Finishing of Aluminum. Walter E. Pocock. Proc. Am. Electroplaters' Soc., **44,** 152-155, disc., 170 (1957); A.S.M. Metals Review, **31,** No. 5, 43 (1958) May.

Chromate treatment can often be substituted for anodizing. It requires no electrical current, short time for processing, no venting for tank, and operates at room temperature. Coating can be applied by immersion, brush or spray; dimensional change is negligible: control of bath is relatively simple.—ALL. 15988

6. MATERIALS OF CONSTRUCTION

6.3 Non-ferrous Metals and Alloys—Heavy

6.3 5

Niobium. Part II. Properties and Applications. J. H. Rendall. Metal Treatment and Drop Forging, **25,** No. 148, 7-12 (1958) Jan.

Presents data on mechanical, physical and thermal properties of niobium, and discusses its application in nuclear reactors (fuel canning material), its use in stainless steels as carbide stabilizer to prevent intercrystalline corrosion, and its use in heat resisting alloys. Known and possible alloys of niobium are considered. Composition, use, and results of creep tests are tabulated for following niobium-containing heat resistant alloys: 326 Firth-Vickers steel, 19-9 tungsten-molybdenum, Gamma-Cb, G. 18 B, Multimet, Inconel X, S.816 and G.32.—INCO. 15497

6.3.5, 3.2.3

The Scaling of Niobium in Air. H. Inouye. Paper before Am. Inst. Mining and Met. Engrs., Regional Conf. on Reactive Metals, Buffalo, March 19-21, 1956. AIME, IMD Special Rept. Series No. 5, 1957, 130-135.

Oxidation rate of niobium was measured at 400, 600, 700, 800, 900 and 1200 C. At all these temperatures except 400 C, rate curves were linear, provided geometry of test specimens was such as to minimize changes in their surface areas. At 400 C, oxide film was protective for first 21 hr. of oxidation, but thereafter rate increased drastically and rate curve became linear. Activation energies of 13,400 and 4,350 cal per mole were found for temperature ranges 600-900 C and 900-1200 C respectively. Change in slope of activation curve occurred at 900 C, temperature at which T modification of niobium pentoxide disappears. Use of niobium in air at high temperatures will necessitate application of a protective coating or development of oxidation-resistant alloys. Unlike molybdenum, which is also being considered for high-temperature application, niobium oxide

was not volatile up to temperatures investigated. Although oxidation rate was high, the rate was one-twentieth that of molybdenum at 800 C. It may be possible to develop alloys of niobium through the use of oxide-oxide reactions. Graphs, photos.—INCO. 15915

6.3.5, 8.4.5

Niobium as a Nuclear Metal. M. J. Cotter. Atomics, **8,** No. 9, 339-342 (1957).

Excellent high-temperature properties, together with a low neutron-cross-section, favors niobium as a canning metal in high-temperature liquid-metal-cooled nuclear reactors. Fabrication methods are described. Uranium-niobium alloys when quenched from the γ phase have excellent corrosion-resistance. Wider application of niobium is limited by its present high price.—MA. 15568

6.3.5, 8.8.5

The Metallurgy of Niobium. M. D. Jepson. Research, **10,** No. 10, 390-395 (1957).

Niobium powder, produced by reaction of niobium pentoxide with niobium carbide or by sodium reduction of K_2NbF_7, is consolidated by sintering cold-pressed compacts in vacuo at 2300 C, the non-metallic impurities being volatilized. The pure niobium thus produced is ductile, and can be formed cold to sheet, tubes, rod, or wire; it can be welded if protected from gaseous contamination. The mechanical properties depend on the purity. Niobium is but little attacked by pure liquid sodium, but the attack is much greater if a little dissolved oxygen is present. The only present industrial use of niobium is as a canning material in nuclear reactors; it is being used in the Dounreay experimental fast reactor. 14 references.—MA. 15911

6.3.6

Cupro-Nickels Offer Corrosion Resistance and Hot Strength. J. L. Everhart. Materials in Design Eng., **47,** No. 5, 114-120 (1958) May.

Cupro-nickels have been used for many years in marine service but increased availability of nickel is reviving and stimulating interest in application of these alloys to other fields. Composed essentially of copper and nickel, the group of commercial alloys ranges in nickel content from 2.5-30; alloys containing 30 and 10 nickel are most widely used. Outstanding attributes of cupro-nickels are corrosion resistance, particularly their resistance to corrosive and erosive effects of high velocity sea water and strength at elevated temperatures. They are widely used in applications requiring exceptional corrosion resistance such as heat exchanger, condenser or evaporator service involving salt solutions. They are also used in applications involving contact with food products. Photos, tables, graphs.—INCO. 16017

6.3.6

New Copper-Base Casting Alloy. K. I. Akimova and A. V. Kurdyumov. Liteinoe Proizvodstvo, No. 2, 10-12 (1958) Feb.

Properties and structures of new copper-base alloy with about 10% each of nickel and aluminum, up to 2% manganese and up to 0.5% iron. Effect of heat treatment on structure and properties is discussed. Good casting, mechanical and corrosion-resisting properties make alloy suitable for wide applications.—INCO. 16038

6.3.8, 4.3.5, 4.3.2

The Corrosion of Lead by Bromine and Its Prevention. M. R. Bloch, D. Kaplan and J. Schnerb. J. Applied Chem., **8,** Pt. 3, 171-174 (1958) March.

Lead corrosion by bromine occurs in presence of hydrobromic acid. It may be obviated by preventing formation of this acid or by removing it from the bromine. Lead dioxide proved to be a satisfactory agent for protecting lead against corrosion by bromine by reacting with any small amount of hydrobromic acid which may have been formed by the action of reducing matter present. Photos.—INCO. 15638

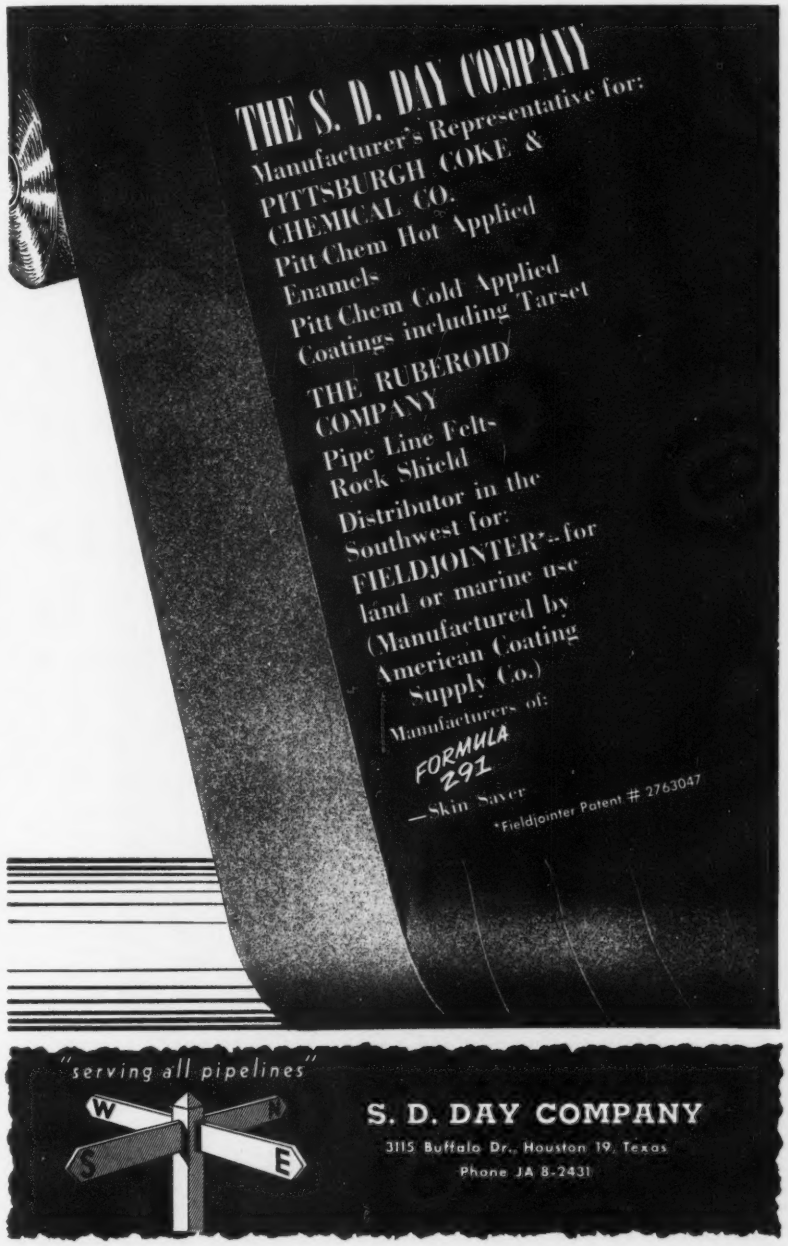
6.3.8, 4.4.2, 3.2.2

The Corrosion of Lead by Dilute Aqueous Organic Acids. E. L. Coles, J. G. Gibson and R. M. Hinde. J. Applied Chem., **8,** Pt. 5, 341-348 (1958) May.

Corrosion tests have been carried out on lead totally immersed in solutions of organic acids (0.1-0.0001 Normal). The corrosion rates in acetic, propionic, butyric, succinic and lactic acids of the same normality were very similar, while pyruvic acid appeared to form a protective film. Intercrystalline attack was the most important general diagnostic feature of the corrosion by the acids, particularly the acetic, propionic and butyric acids. X-ray diffraction analysis has established that the corrosion products in most cases gave patterns which could be arranged in a series showing a gradual transition from basic carbonates deficient in carbon dioxide to the basic carbonate $2PbCO_3, Pb(OH)_2$.—BTR. 16025

6.3.10, 4.7

Corrosion Resistance of Nickel Alloys in Molten Sodium Hydroxide. H. B.



Probst, C. E. May and Howard T. McHenry. U. S. National Advisory Committee for Aeronautics, Technical Note 4157, January, 1958, 26 pp.

The corrosion resistance of 11 noncommercial solid-solution alloys and two-phase materials at 1500 and 1700 F was compared with the excellent corrosion-resistant properties of pure nickel. The resistance of the nickel plus 30% copper alloy was comparable to that of pure nickel. Other materials that appeared promising were nickel plus molybdenum, nickel plus tin, nickel plus TiC, nickel plus magnesium oxide and nickel plus aluminum oxide. 15473

6.3.10, 8.9.5

Nickel and Its Non-Ferrous Alloys in Shipbuilding. L. Arbellot. Rev. Nickel, **23**, No. 3, 73-85 (1957).

A survey of applications of nickel-copper alloys such as Monel, cupro-nickels, nickel bronzes, aluminum bronze with nickel and nickel-chromium alloys is given, with a brief account of the iron-nickel chromium stainless alloys. Nickel-plated steel is also referred to. The composition and mechanical properties of Monel are shown, with Hastelloy and Inconel, including hardness, fatigue and corrosion-resistance, and their various uses in ships are noted, including steam gaskets, expansion joints and centrifugal pump parts, also Monel welding rods. Cupro-nickels are similarly treated for condenser tubes, with special reference to corrosion-resistance, as are the nickel-bronzes and brasses and copper-aluminum-nickel compositions, with special reference to propellors and cavitation and erosion effects. Nimonic 100 in gas-turbines is also reported.—MA. 16131

6.3.10, 4.3.2

The Corrosion of Monel and 70-30 Cupronickel in Hydrofluoric Acid. Walter J. Braun, Frederick, W. Fink and G. Lee Ericson. Battelle Memorial Inst. U. S. Atomic Energy Commission Pubn., BMI-1237, December 3, 1957, 21 pp. Available from Office of Technical Services, Washington, D. C.

Monel is shown to be suitable for the handling of 10 to 70% hydrofluoric acid at 140 F and for handling 38 and 48% acid at boiling; cupronickel also is useful over part of the this range. Elimination of oxygen is necessary to achieve maximum corrosion resistance. Some accelerated interface attack was noted for 70-30 cupronickel. Welding of Monel is the preferable joining method. Silver solder joints are subject to embrittlement. Work hardening, annealing and external stress do not significantly influence the corrosion of Monel. Galvanic coupling of Monel with silver or silver solder is to be avoided. (auth.)—NSA. 15713

6.3.10, 7.1

Research on Nickel-Chromium and Nickel-Chromium-Cobalt Alloys for Gas-Turbine Rotor Blades: Practical Results. J. Poulignier. Rev. Nickel, **23**, No. 1, 1-8 (1957); ibid., No. 2, 40-44 (1957).

The following aspects are reviewed and photomicrographically illustrated: metallographic examination of the blades after use; study of the surfaces corroded by combustion gases; assessment of temperatures reached by various parts of the blades; cold-working and surface-recrystallization phenomena. 14 references.—MA. 15982

6.3.17

Preliminary Report on Low Niobium. Uranium Corrosion Resistant Alloys. J. E. Draley. Argonne National Lab. U. S. Atomic Energy Commission Pubn., ANL-5078, June 24, 1953 (Declassified April 2, 1957), 9 pp. Available from Office of Technical Services, Washington, D. C.

Uranium alloys containing 3 and 6 wt.% niobium and 1.5% niobium-1.2% zirconium have been prepared in which the impurity content is low. These alloys show very promising resistance to corrosion by water at 260 and 300 C. The corrosion resistance is exhibited in degassed water and in water which is saturated with air or oxygen at room temperature before testing. (auth.)—NSA. 15763

6.3.17, 3.7.2

Alloys of Uranium and Silicon. Pt. II. The Epsilon Phase. Gust Bitsianes. Massachusetts Inst. of Tech. U. S. Atomic Energy Commission Pubn., CT-3309, June 1945 (Declassified Feb. 16, 1957), 32 pp. Available from Office of Technical Services, Washington, D. C.

Epsilon (ε) is an intermediate phase in the uranium-silicon system containing about 23 at% silicon. This phase forms by means of a peritectoid reaction whose temperature is lowered by the presence of carbon. Investigation of the corrosion resistance of ε in various media shows that ε is quite superior to pure uranium metal and many other uranium alloys. The ε phase is a high-strength alloy and possesses some degree of ductility. These properties indicate a distinct possibility of using ε in direct contact with cooling water. (auth.)—NSA. 15732

6.3.17, 4.6.2

Corrosion Mechanism of Uranium-Base Alloys in High Temperature Water. M. W. Burkart and B. Lustman. Westinghouse Electric Co. Trans. Metallurgical Soc. AIME, **212,** No. 1, 26-31 (1958) Feb.

Uranium-base alloys exposed to high temperature water fail either by uniform oxidation or by sudden cracking and disintegration of metal. Disintegration results from oxidation of a second phase which is precipitated during corrosion process. Precipitate has been identified as a metastable hydride of uranium. Alloys used in investigation were γ phase uranium alloys containing 7-12 molybdenum or niobium. Tests were conducted in 650 F water. Graphs, photomicrographs.—INCO. 15462

6.3.17

Results of Natural Uranium (3.8 wt.%) Zirconium Alloy; DIG-CWA Corrosion Tests. Progress Report No. 1. D. C. Belouin, E. G. Brush and R. E. Campagnoni. Knolls Atomic Power Lab. U. S. Atomic Energy Commission Pubn., KAPL-M-EGB-26, December 1957, 20 pp. Available from Office of Technical Services, Washington, D. C.

Tests have been conducted in deionized water at 200, 400 and 600 F to determine uranium release through corrosion of 4 wt% uranium-zirconium alloy, both coupled and uncoupled with 2S aluminum at 200 F, uncoupled only at 400 and 600 F. Results indicate that uranium release of uncoupled uranium-zirconium up to 1500 and 1000 hours, respectively in water at 200 and 400 F, is about 5 x 10^{-5} mg/dm²/hr. At 600 F somewhat more severe corrosion occurs with formation and spallation of characteristic yellow corrosion product (absent at the lower temperatures). Galvanic coupling of uranium-zirconium with 2S aluminum results in increased corrosion of the aluminum through differential aeration cells rather than by galvanic attack in 200 F water. The results also indicate that, at constant temperature (200 F) the corrosion rate increases with oxygen contamination in the test medium. Furthermore there appears to be a change in corrosion mechanism with temperature so that room temperature corrosion rates obtained by extrapolation may vary approximately by a factor of 10. Further room temperature tests are underway to narrow this spread. (auth.)—NSA. 15541

6.3.20, 3.2.3, 3.7.2

Scaling of Zirconium and Zirconium Alloys. Technical Progress Report No. 1. J. A. Burka, C. S. Crouse and R. E. Swift. Kentucky Univ. U. S. Atomic Energy Commission Pubn., AECU-3561, August, 1957, 46 pp. Available from Office of Technical Services, Washington, D. C.

Zirconium-base binary alloys were prepared using the elements molybdenum, iron, cobalt, chromium, nickel, copper, silver, tantalum, niobium, platinum, protactinium, osmium, iridium, rhodium, ruthenium, vanadium, silicon, manganese, tin, lead, tungsten, antimony, germanium, aluminum, titanium, magnesium, zinc, lanthanum and barium. Also, zirconium-base ternary alloys were prepared using the elements copper, nickel, niobium, tantalum, vanadium, tin, antimony and tungsten. The maximum total alloy content of the alloys was five per cent. The zirconium used was low-hafnium sponge and the alloys were prepared by arc melting in an argon atmosphere. The alloys were tested for scaling resistance at the temperatures 1200, 1500 and 1800 F in a multi-specimen furnace for periods up to about 300 hours. Scaling propensity of the alloys was evaluated on the basis of the weight increase per unit area as compared to that of unalloyed zirconium. The binary alloys containing nickel and copper showed the greatest scaling resistance of all the binary alloys at all temperatures. Ternary alloys showed little or no improvement over the most scaling resistant binary alloys. The number of alloys more resistant than unalloyed zirconium was about the same at 1200 and 1500 F, but was considerably more at 1800 F. In general, there was some change in the order of relative scaling propensity based on the average weight increase per average unit area per hour at the three temperatures. A logarithmic plot of weight increase versus time for most of the alloys showed a sharp increase in the scaling rate after various periods of time, whereas very few showed a decrease. (auth.)—NSA. 15745

6.3.20, 2.3.4, 4.6.2

Comparison of 680 F Water and 750 F Steam as an Acceptance Test for Zircaloy. A. B. Riedinger. Knolls Atomic Power Lab. U. S. Atomic Energy Commission Pubn., KAPL-M-ABR-3, Feb., 1958, 14 pp. Available from Office of Technical Services, Washington, D. C.

From these results it is indicated that a statistically significant correlation can be obtained from the results of three-day, 750 F steam and fourteen-day, 680 F water tests. The results of these two

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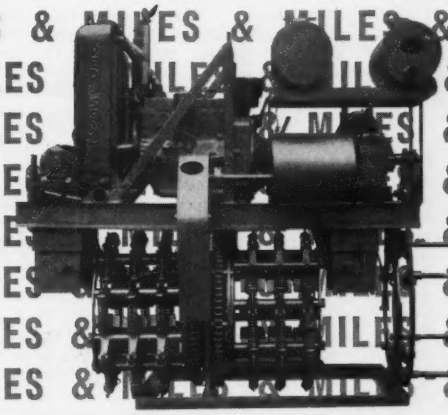

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tests show sufficient differences so that they cannot be considered interchangeable. Short-term tests, such as one, three, or fourteen days in 750 F steam or fourteen days or less in 680 F water can only be expected to detect gross variation in corrosion resistance. (auth.) —NSA. 16081

6.3.20, 8.4.5

Zircaloy Tube for Nuclear Power. J. S. Rodgers. Calumet, Hecla, Inc. Can. Metalworking, **21,** No. 4, 24, 26 (1958) April.

Advent of atomic age has brought along with it an increased need for zirconium and zirconium alloys (Zircaloys), metals that, although difficult to work, are of great importance as structural materials in the atomic energy field. Zircaloys are used in atomic reactors because of their low neutron absorption, good corrosion resistance and high melting temperature. In certain nuclear power plants, such as those on ships, they are virtually irreplaceable because they do not "poison" the uranium fuel as rapidly as less expensive metals. Fabrication of Zircaloys is described. Photos.—INCO. 16095

6.3.20

Certain Properties of Zirconium-Niobium Alloys. In. F. Bychkov, A. N. Rozanov and D. M. Skorov. Atomnaya Energ., **2,** 146-151 (1957). U. S. Atomic Energy Commission Pubn. Translations, AEC-tr-3133. Available from SLA Translation Pool, John Crerar Library, 86 E. Randolph St., Chicago 1, Illinois.

A phase diagram of the zirconium-niobium system constructed from experimental data is presented. Results are given of the expansion of zirconium-niobium alloys at room temperature and an estimate of their hot strength from hardness measurements at temperatures up to 750 C, and a measurement of the hardness of alloys resulting from low temperature annealing (aging) is also given. The data obtained are explained using the phase diagram. The heat resistance of alloys oxidized in the air at 570 and 650 C is determined. (auth.) —NSA. 15560

6.3.20, 1.7.1, 3.7.2

Report of the March 1953 Meeting of the Zirconium Alloy Corrosion Committee. K. M. Goldman. Westinghouse Electric Corp. U. S. Atomic Energy Commission Pubn., WAPD-MM-200 (Del.) March 24, 1953 (Declassified with Deletions March 5, 1957), 68 pp. Available from Office of Technical Services, Washington, D. C.

The results of work done on the corrosion of Zircaloy-2 and other alloys are presented. Zircaloy-2 shows excellent corrosion resistance to steam at 750 F and water at 680 F for periods up to three and four months, respectively. There is little or no effect of tin content on the corrosion resistance of sponge-base alloys of zirconium containing 0.12% iron, 0.10% chromium and 0.05% nickel tested for 45 days at 750 F and 28 days at 680 F. This suggests lowering the tin content in Zircaloy-2 to obtain increased fabricability with no sacrifice in corrosion resistance. Enough tin should remain to insure a good nitrogen tolerance. Although relative effectiveness of iron, nickel and chromium in Zircaloy-2 has not yet been determined, it appears that low totals of iron, nickel and chromium are detrimental to corrosion resistance. Some samples of unalloyed zirconium sponge displayed excellent corrosion resistance to 680 F water for 14 days and 750 F steam for 48 hours.—NSA. 15789

6.4 Non-ferrous Metals and Alloys—Light

6.4.2

High Heat Resisting Aluminum. R. Irmann. *Can. Metalworking,* **20,** No. 12, 38-40, 65 (1957) December.

Presents up-to-date information on sintered aluminum product SAP, covering mechanical properties, physical and thermal properties, corrosion behavior, formability, forging, machining, riveting, welding and applications. Drop forged compressor wheel and compressor blade are shown. Graph compares creep-rupture properties at 540 C for SAP, pure titanium and titanium alloys.—INCO 15335

6.4.2, 1.3

The Production and Properties of Super-Purity Aluminum. T. G. Pearson and H. W. L. Phillips. Metallurgical Reviews, **2,** No. 8, 305-360 (1957).

Comprehensive review of super-purity aluminum covers: electrolytic refining by 3-layer process, methods for analysis for certain impurities (including nickel), physical properties, mechanical properties (including high temperature properties), physical metallurgy, properties of zone-refined super-purity aluminum, chemical properties (includes corrosion data), fabrication and joining. 178 references.—INCO. 15922

6.4.2, 3.6.6

The Corrosion Behavior of Industrial Aluminum in Weathering and in Contact with Other Metals. (In German.) H. M. Cohen. Mitt. Forschungsgesellschaft Blechverarbeitung, **19,** 213-223 (1957); Aluminium, **34,** No. 2, A36 (1958) Feb.

Results of a number of tests carried out on the corrosion behavior of aluminum and aluminum alloys, as compared with other metals. Tests were conducted under various climatic and industrial conditions, such as salt mining, soda production, petroleum refining, etc., complemented with numerous service records relative to aluminum roofing. The galvanic corrosion process taking place in bimetallic couples is discussed and recommendations are given as to the corrosion protection of aluminum by using oxide films, paint or electric insulation.—ALL. 15555

6.4.2, 4.6.5, 3.8.4

Corrosion of Aluminum in Deionized Water. Progress Report for September 1957-December 1957. J. A. Ayres and R. L. Dillon. General Electric Co., Hanford Atomic Products Operation. U. S. Atomic Energy Commission Pubn., HW-53963, December 6, 1957, 9 pp. Available from Office of Technical Services, Washington, D. C.

Static and dynamic tests were conducted to determine the effects of flow, hydrogen ion concentration, common ion effect and inhibitors on corrosion rates of aluminum. Autoclave studies were designed to determine rates and mechanism of corrosion.—NSA. 15716

6.4.2, 3.7.2, 8.4.5

New Aluminum Alloy Developed. Electrical News and Engineering, **67,** No. 5, 61 (1958) May.

The Argonne Laboratory has developed an aluminum alloy, designated X-8001, for nuclear fuel element cladding. Aluminum has been considered unsuitable for use in reactors because of its tendency to form a crystalline structure at relatively low temperatures. Alloy X-8001 was developed by adding small amounts of nickel to aluminum 1000, a commercial alloy containing iron. The nickel addition made X-8001 much more resistant to corrosion by high temperature water than commercial aluminums having iron as the primary alloy material. Fuel elements clad with this alloy are ideal for use in small reactors. —ALL. 15950

6.4.2, 5.9.2

Chemical Protection Against Oxidation, of Aluminum and Its Alloys Before Coating. (In French.) Claude Hess Corrosion et Anti-corrosion, **6,** No. 2, 44-47 (1958) Feb.

Causes for the increasing use of aluminum and aluminum alloys. Types of alloys having a high mechanical or corrosion resistance. Electrochemical and chemical surface treatments. Exposure tests.—BTR. 15684

6.4.2, 4.6.11, 3.7.2, 8.9.5

Corrosion of Aluminum Alloys Used for Ship Parts. M. Orman. Prace Inst. Ministerstwa Hutnictwa, **9,** 167-171 (1957); Chem. Absts., **52,** No. 4, 2714 (1958) Feb. 25.

Orman suggests addition of chromium, titanium or vanadium to the aluminum-magnesium alloys to prevent the intercrystalline corrosion of these alloys in sea water and sea atmosphere. It is shown that aluminum alloys containing magnesium 2.5-3.5%, chromium 0.15-0.39%, manganese 0.15-0.35%, silicon maximum 0.4%, iron maximum 0.4%, copper 0.05%, zinc 0.05%, and silicon + iron + copper + zinc 0.7% are perfectly resistant to corrosion by sea water. Aluminum-manganese alloys, however, cannot be used for external ship parts. —ALL. 15923

6.4.2, 8.3.1, 4.3.4

Applications of Aluminium in Agriculture. A. Perrone. Alluminio, **26,** No. 5, 216-219 (1957).

The high corrosion resistance and other favorable properties of aluminum recommend it for agricultural uses. Examples of application are shown. Resistance to superphosphate and other fertilizers is compared with that of galvanized sheet.—MA. 15898

6.4.2, 8.9.1, 1.7.1

Aluminium Alloys in Aircraft Structures. G. Meikle. North Atlantic Treaty Organization, Paris. Advisory Group for Aeronautical Research and Development, April, 1957, 15 pp.

Presented at the Fifth Meeting of the Structures and Materials Panel, April 24 to 27, 1957, in Oslo, Norway. Details of compositions of three groups of aluminum alloys used in the United Kingdom are discussed in relation to alloys used in the U.S.A. The properties of these alloys at elevated temperatures are mentioned particularly with regard to the effect of time and temperature, this being of great importance where kinetic heating is involved. The different nominal compositions of the high strength aluminum zinc-magnesium alloys used in the United Kingdom are dealt with, and some of the difficulties experienced with stress-corrosion in this type of alloy are mentioned. Research at the Royal Aircraft Establishment on

this program is briefly mentioned with particular reference to the effect of chemical composition and heat treatment. Brief reference is made to a possible aluminum alloy for use at temperatures up to 200 C. (auth.)—NSA. 15963

6.4.4, 5.9.4

Some New Data on the Protection of Magnesium Based Alloys. (In French.) W. F. Higgins. Corrosion et Anticorrosion, **6,** No. 2, 40-43 (1958) Feb.

Importance of protection for magnesium parts. Anodic treatment: correct drawing, surface purification and passivation, sealing, correct assembling and final coating.—BTR. 15630

6.4.4, 3.8.4

Kinetics of the Gas Corrosion of Magnesium Alloys. (In Russian.) I. A. Makolkin. *J. Applied Chem. USSR* (Z. Prikladnoi Khim.), **30,** No. 10, 1542-1547 (1957) October.

Study of gas corrosion of magnesium alloys ML-4 and ML-5 at temperatures below 400 C and of alloy MA-1 in the range from 450-600 C—BTR. 15364

6.4.4

Magnesium and Its Alloys and Their Uses. (In French.) M. Hardouin. Usine nouvelle, Special No., 87-89, 91 (1957).

Production and properties, sources and output are summarized and minor uses noted, such as a reducing agent in metallurgy and in nuclear piles. Alloys for casting and welding and the newer, more resistant composition are mentioned and typical parts for various industries are shown. Use in printing and cathodic protection.—ALL. 15858

6.4.4, 3.7.2

Magnesium Alloys with High Melting Point Additions. R. R. Nash and others Rensselaer Polytechnic Institute for U. S. Wright Air Development Center, U. S. Air Force, March, 1957, 196 pp. Available from Office of Technical Services, U. S. Dept. of Commerce, Washington 25, D. C. (Order PB 131198.)

Presented are data and a number of conclusions drawn from an exploratory investigation to determine the alloying characteristics and the influence on microstructures, mechanical properties and corrosion resistance of additions of high melting point elements to magnesium and magnesium-base alloys. Additions were titanium, hafnium, tantalum, nickel, cobalt, vanadium, boron, chromium, yttrium, rhenium, niobium, tungsten and molybdenum. The study was part of a project aimed at development of new wrought magnesium-base alloys with mechanical and physical properties better than those of present-day commercial alloys.—OTS. 15881

6.4.4, 3.8.4

A Basic Study of Corrosion of Magnesium. R. R. Addiss and others. Cornell University for Wright Air Development Center, U. S. Air Force. December, 1957, 57 pp. Available from Office of Technical Services, U. S. Dept. of Commerce, Washington, D. C. (Order PB 131662).

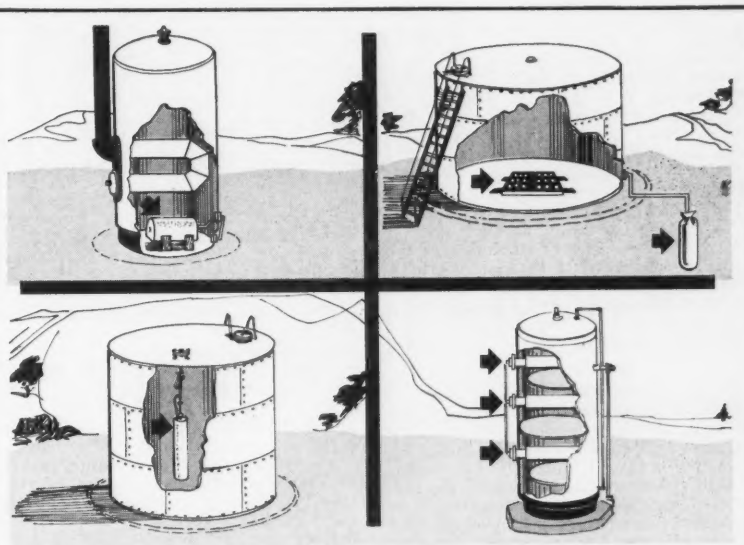
This project was intended to expand understanding of the process of corrosion of magnesium and its alloys through basic study of the structure of the surface of the metal and of the process of formation and structure of surface films appearing during corrosion. A number of attempts were made to obtain a perfectly clean surface, both by cleaning a contaminated surface and by producing a new, clean surface in vacuum. One attempt with the vacuum approach met with partial success. What was believed to be a clean magnesium crystal was studied by a field emission microscope. Studies were also made of oxidation of surfaces covered with the first film. These were aimed at measuring the oxidation rate to obtain insight into the relative crystallographic orientation of the metal and the oxide.—OTS. 15727

6.7 Duplex Materials

6.7.2

Cermets. (In Italian.) C. A. Bertella. Metallurgia Italiana, **49,** No. 8, 637-646 (1957).

Bertella reviews recent progress, including methods of testing cermets. In laboratory research, temperatures up to 1825 C have been attained in a molybdenum furnace and up to 1960 C in a graphite furnace. Tests described are for creep, thermal shock, corrosion and thermal expansion, as well as micrographic examination and practical tests. Chromium-Al_2O_3 cermets give high U.T.S. and creep-resistance up to 1200 C. Silicon-titanium dioxide cermets are being investigated. Titanium carbide-base cermets containing other carbides and with a metal binder (cobalt, nickel-chromium or nickel-chromium-cobalt) give high corrosion-resistance. Other carbide cermets are zirconium carbide-niobium and chromium carbide-nickel. Boride cermets have high hardness and good resistance to creep, thermal shock and corrosion. 18 references.—MA. 15709



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6.7.2, 5.3.4

Status of an Alternate Control Materials Program. R. W. Dayton. Battelle Memorial Inst. U. S. Atomic Energy Commission Pubn., BMI-X-134, December 15, 1956 (Declassified Feb. 14, 1957), 14 pp. Available from Office of Technical Services, Washington, D. C.

Mechanical properties of clad and unclad boron-titanium and B_4C-Ti cermets and the corrosion resistance of defected clad compacts of these combinations was investigated. Primary emphasis was placed upon a 26 wt% boron in titanium despersion and a 35 wt% B_4C in titanium dispersion. One compact of each of these compositions was prepared by hot pressing green compacts sealed in mild steel frames at 1500 F. These specimens are being machined and will be clad by pressure bonding techniques. After cladding the specimens will be defected and corrosion tested in 680 F water. (auth.)—NSA. 15908

6.7.2, 7.1

Experimental Investigation of Cermet Turbine-Blades in an Axial-flow Turbojet Engine. William C. Morgan and George C. Deutsch. U. S. National Advisory Cttee. Aeronautics, Technical Note No. 4030, 1957, 20 pp.

K-152B (titanium carbide 62—titanium, tantalum, niobium—carbon 8, and nickel 30%) cermet blades were tested in an engine operating at 7950 rpm at 1260 F (680 C). Lives of 150 hr were achieved in the most satisfactory tests with only minor chipping of the blades. Primary cause of failure was the fracture of the airfoil. The effect of blade design was studied.—MA. 15870

6.7.2

What's Happened to Cermets? J. W. Graham and W. F. Zimmerman. General Electric Co. Metal Progress, **73,** No. 3, 89-91 (1958) March.

A brief summary is given of the history of development of cermets. The oxidation, mechanical properties and impact resistance are reviewed. Possible future applications are discussed.—NSA. 16018

6.7.2, 8.9.1

What Can Ceramics do in Missiles? J. Castelfranco. North Am. Aviation Inc. Ceram. Ind., **70,** No. 5, 84-89 (1958) May.

A variety of ceramic bodies are used as liners for uncooled motors and exhaust nozzles and for protection of metallic structures at stagnation points. Cermets endeavor to combine desirable properties at elevated temperatures of ceramics and metals and have found successful application in many missile components such as valves, pumps and bearings. Nevertheless 1800 F should be regarded as maximum temperature at which cermets prove useful for missile applications. Photos show use of titanium carbide cermets (potassium-162-boron-25 nickel, 5 molybdenum, 64 titanium carbide and 6 niobium carbide) for fabrication of self-aligning bearings that have to operate at 1500 F. Refractory coatings are used as protection of metallic type structures for short time applications. Impervious coatings make possible the utilization of molybdenum on a wider range of temperatures. Impervious and abrasive coatings allow utilization of graphitic materials at stagnation points without deterioration of aerodynamic configurations. Emissive coatings lower considerably the equilibrium temperatures of bodies flying at high Mach numbers and at great altitudes. Diagrams, photos.—INCO. 16041

6.7.2

New Alumina-Type Cermets. Thomas F. Frangos. Materials in Design Engineering, **47,** No. 2, 112-115 (1958) Feb.

These cermets retain useful strengths at temperatures above 2300 F, and have excellent resistance to molten metals (except aluminum), chemicals, oxidation, wear and high temperature erosion. The products are produced by slip casting, permitting considerable design flexibility. Finished shapes to close tolerances can be produced by machining and grinding. They are relatively brittle and not recommended for use under high stress or mechanical shock conditions; they should not be used in carburizing atmospheres, since they become more brittle and there is some dimensional distortion, and they are not recommended for use in acid or carbide slags. Three types having alumina as the ceramic phase have been developed: LT-1, the first type, consists of 77 chromium and 23% aluminum oxide by weight. It has excellent resistance to oxidation at temperature up to above 2200 F. It resists wetting by molten metals, is chemically inert to many corrosives, and has moderate strength at temperatures where many metals become molten. In LT-1b, molybdenum and a small percentage of titanium oxide are substituted for part of the chromium and alumina content. This provides an increase in tensile strength and stress-rupture properties, as well as thermal shock resistance. There is a slight loss in oxidation resistance. In LT-2, tungsten is substituted for a considerable part of the chromium content which further improves strength and resistance to abrasion and erosion. It improves modulus of rupture, erosion resistance and hardness but at considerable sacrifice to oxidation resistance. Photographs illustrating these cermets include mechanical seals used in transfer pumps for high concentration sulfuric acid or oleum, flame holders, rocket nozzle inserts, thermocouple protection tubes, for open hearth furnaces, flow control pins, for controlling flow of molten copper in a continuous casting machine, and a T-shaped pouring spout, to feed copper from a continuous casting machine to a rectangular ingot mould.—ALL. 15449

7. EQUIPMENT

7.1 Engines, Bearings and Turbines

7.1, 5.8.2, 8.9.4

Copper-Lead Bearing Corrosion Inhibition in Diesel Service. J. R. Thomas, O. L. Harle, W. L. Richardson and L. O. Bowman. Paper before Am. Chem. Soc., Petroleum Chemistry Div., 130th Ann. Mtg., Atlantic City, Sept. 1956. *Ind. & Eng. Chem.*, **49,** No. 10, 1703-1708 (1957) October.

Describes intensive research which led to development of class of inhibitors suitable as oil additives for use with both copper-lead and silver bearings. Mechanisms of copper-lead bearing corrosion, corrosive conditions in GM 3-71 engine, mechanisms of corrosion inhibition and factors determining effectiveness of protective film-forming inhibitors are discussed. Inhibitors developed effectively inhibit corrosion of copper-lead bearings; at same time, oils containing these materials effectively lubricate silver bearings and are not corrosive to them. Inhibitors were designed to be acidic and are capable of reacting with lead oxide to give tough refractory films. Their acid strengths were selected so that their acid salts are basic to sulfuric acid and oxy-acids in order not to impair detergent and antiwear properties of basic additives. Experimental test procedures are outlined. Graphs.—INCO. 15125

7.1

First Commercial Supercritical-Pressure Steam Turbine: Built for the Philo Plant. C. W. Elston and R. L. Shepard. Paper before Am. Soc. Mech. Engrs., Ann. Mtg., Chicago, Nov. 13-18, 1955. *Trans. ASME*, **79,** No. 2, 417-426; disc., 426 (1957) Feb.

Essential design features of 125,000-kw, 36,000-rpm, tandem-compound, double-flow steam turbine designed for steam conditions of 4500 psig, 1150 F initial temperature (double reheat). Thermal stresses are minimized by keeping wall thicknesses of austenitic steel parts as small as possible; this was accomplished by use of steam cooling, individual control-valves of small diameter, multiple steam pipes and simple throttling control. Thermodynamics involved are discussed. Outer and inner casings of high-pressure turbine section are cast chromium-molybdenum-vanadium ferritic steel; inner casing is lined with Type 347. High-pressure rotors are of three types: all austenitic (solid forging of Type 347 with buckets of 12 chromium-1 molybdenum-1 tungsten-¼ vanadium steel), steam-cooled ferritic (1 chromium-1¼ molybdenum-¼ vanadium steel) and composite rotor in which Type 347 section is welded directly to ferritic chromium-molybdenum-vanadium sections. Bolting is 19 chromium-12 nickel-3 tungsten -1 niobium steel for austenitic parts, and 12 chromium for ferritic parts. Piping and valves are of Type 347. Electrodes for welding are low-ferrite Type 347 and 16-8-2 chromium-nickel-molybdenum (Babcock & Wilcox). Valve-stem packings are soft rings of graphitized asbestos reinforced with nickel alloy. Schematic diagrams. —INCO. 14959

7.2 Valves, Pipes and Meters

7.2, 6.6.5

Pressure Concrete Pipe Handles Corrosives. R. B. Norden. *Chem. Eng.*, **64,** No. 8, 300, 302 (1957) August.

New design of concrete pipe, now in use at Dow Chemical's Texas division, is capable of withstanding 50-300 psi. Precast pipe consists of sections each having a steel cylinder embedded in dense concrete. Pipe is joined by exposing and welding end sections of steel cylinders. Pipe is prestressed during manufacture by wrapping under tension high tensile steel wire around steel cylinder, thus placing steel and concrete under compression. Most of pipe at Dow handles sea water and cooling water, but use for neutral and alkaline chemicals is being considered. —INCO. 15047

7.2, 8.9.1

Choosing Valve Materials. J. R. Driear. Eaton Mfg. Co. *Steel*, **141,** No. 24, 161-162 (1957) Dec. 9.

An aircraft type poppet valve is made Inconel M base; tool steel tip is buttwelded on and hardened for wear; stem is coated with 0.005-in. layer of AMS-4775 (nickel-chromium-boride) for wear resistance;

and top of head and seat are coated with chromium-nickel-tungsten alloy. Design and selection of materials for inlet valves and exhaust valves are discussed. Table lists uses of following poppet valve materials: Silchrome No. 1, Silchrome XBe, Silchrome 10, Silchrome 142, 21-4N, 21-12N, N-155, Inconel M, Nimonic 90, Bright Ray, Eatonite, AMS-4775 and a chromium-nickel-tungsten alloy.—INCO. 15212

7.2, 2.3.7

Coiled Stainless Steel Tubing: Part 1. Test Results. J. J. Chmura, Jr., R. D. Stouffer and A. N. Winter. The Martin Co. for U. S. Wright Air Development Center, U. S. Air Force, Dec., 1957, 136 pp. Available from Office of Technical Services, U. S. Dept. of Commerce, Washington, D. C. (Order PB 131719).

Fatigue characteristics of various sizes of coiled stainless steel tubing and torsional tubes were determined. Aims were to determine the thinnest walled tube for selective diameters capable of withstanding 200,000 cycles, and to find the greatest deflection obtainable for these tubes. Results led to the recommendation of seamless ¼ hard stainless steel tubing as best for coiled applications. Conclusions were also drawn from tests of flatness, prepressurization, vibration, plumbing connections, configurations, and temperature. Test results were very consistent. The data confirmed analytical procedures established previously for determination of effects of variables on fatigue life.—OTS. 15569

7.4 Heat Exchangers

7.4.2, 3.6.9

Electrolytic Corrosion in Condenser Tubes of a Condensation Turbine. (In German.) F. E. Sell. *Energietechnik*, **7**, No. 9, 413-417 (1957) Sept.

Material and construction of the condenser. Spectral and quantitative examination of tubes composed of 71.8% copper, 27.5% zinc and 1.05 tin. Electrolysis by stray currents; physical and electrical phenomena caused by local elements. Preventive measures.—BTR. 15090

7.4.2, 6.3.6

Resistance to Corrosion of Condenser Tubes Made of Copper Alloys. K. Eichhorn. Werkstoffe u. Korrosion, **8**, No. 11, 657-668 (1957) Nov.

In considering basic electrochemical processes of corrosion in connection with condenser tubes, normal potential, heat of oxidation, passivation and immunity, local cell formation and properties of protective films are discussed. Forms of corrosion include uniform attack, corrosion-erosion, dezincification, deposit attack, stress-corrosion cracking and corrosion fatigue. Degree of wear depends on composition and temperature of steam, the cooling water, method of operation and construction of the condenser. Behavior of cooling water on condenser alloy is chiefly determined by pH of the water and its carbonate, chloride and oxygen contents. Most difficulties are caused by contaminated water especially when condenser is first in operation. Material must be selected according to conditions of operation. For inland waters SoMs 71 (Admiralty brass) and for sea water SoMs 76 (aluminum brass) and 70 copper-30 nickel-iron are best suited. 90 copper-10 nickel and 80 copper-20 nickel are included in evaluation of various coper alloy condenser materials. Cathodic protection is discussed. Photographs and photomicrographs show formation of passivating film and various forms of corrosion. Tables, graphs, 35 references.—INCO. 15773

8. INDUSTRIES

8.4 Group 4

8.4.5

Sodium Graphite Reactor Quarterly Progress Report for January-March, 1957. R. W. Dickinson, Editor. North American Aviation, Atomic International Division. U. S. Atomic Energy Commission Pubn., NAA-SR-1941, July 1, 1957, 54 pp. Available from Office of Technical Services, Washington, D. C.

Criticality and nuclear parameter calculations were initiated for edge-loaded hexagonal moderator core configurations. A cost comparison was made of sodium heat transfer systems using stainless steel and chromium-molybdenum alloy. Loading experiments on the SRE indicate the dry, room temperature critical loading is 22.2 fuel elements. Flux maps were made of the dry, subcritical core. Fuel specimens from the MTR were examined. The examination results substantiated previous data on metal swelling under hot irridiation. Uranium slug casting in thin-walled molds using pressure and static casting techniques was satisfactorily demonstrated. Static and dynamic corrosion of beryllium by liquid sodium was measured. The performances of Mark I and Mark II control rods were compared.—NSA. 15777

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8.4.5

Control Rod Materials. D. N. Dunning and W. E. Ray. Knolls Atomic Power Lab. Nucleonics, **16,** No. 5, 88-92 (1958) May.

Mechanical and corrosion-resistance properties of unirradiated control materials (including stainless steels, rare-earth oxide cermets, boron-titanium, boron-zirconium, hafnium, and cadmium-indium-silver) are tabulated. Only materials considered satisfactory for water-cooled power reactors, other than hafnium are stainless-steel based systems, other materials being applicable to specialized systems. Creep strength of cadmium-indium-silver alloy is low at reactor-operating temperatures due to its low recrystallization temperature. Radiation-damage resistance of rare earth oxide-stainless steel dispersions was studied. In europium oxide-ss dispersions considerable ductility is lost due to irradiation. Cause of this damage is not definitely known, although excessive internal temperatures may have caused an interaction with silicon contained in Type 302. Cladding would add strength to components and would provide a barrier between reactor coolant and europium oxide particles. Gadolinium oxide-ss dispersions exhibited little damage. In study of radiation-damage resistance of boron alloys and dispersions, stainless-steel-boron alloys were found to be most promising if reasonable burnups are allowable, while boron dispersions in titanium are most promising from a damage standpoint. Fabrication of boron, hafnium and other control rod materials is described. Photomicrographs, tables.—INCO. 16015

8.4.5

Recovery of Plutonium and Fission Products from Reactor Pot Residues of the Chlorine Trifluoride Process. S. T. Benton and R. A. Gustison. Carbide and Carbon Chemicals Co. U. S. Atomic Energy Commission Pubn., K-817, Sept. 12, 1951 (Declassified Feb. 23, 1957), 16 pp. Available from Office of Technical Services, Washington, D. C.

Preliminary investigations have shown that the plutonium and fission-product fluoride residue can be recovered quantitatively from the reactor pot of the CTF Process without prohibitive corrosion to the material of construction. Aluminum nitrate was found to be superior to Versene as a solvent in removing the plutonium from a prototype reactor, but Versene more thoroughly removed the fission products, especially ruthenium. The maximum solubility of PuF_4 was 23.2 g/l in 5% $Al(NO_3)_3$ at pH 0.5 and 4.1 g/l in 1% Versene at pH 9. Monel was satisfactory as a material of construction in either $Al(NO_3)_3$ of Versene with a rate of corrosion of 0.022 mils/day and 0.001 mils/day respecively. Data indicates that nickel would not be very satisfactory for use at the optimum solubility conditions of 5% $Al(NO_3)_3$, since the rate of corrosion was 0.366 mils/day. (auth.)—NSA. 15729

8.4.5

Reactor Materials. D. O. Leeser. Atomic Power Development Associates. Nucleonics, **15,** No. 9, 137-139 (1957) Sept.

Technological advances made in the development of reactor materials during the past ten years are reviewed. It is predicted that during the next ten years designers will specify more stringent conditions requiring better thermal, radiation, and corrosion stability. Problems and approaches to their solution are discussed.—NSA. 15992

8.4.5

Chemical Aspects of Nuclear Power. Pt. 2. Reactor Material Problems. F. S. Martin. Atomics, **8,** 168-173 (1957) May.

Chemical aspects of non-metallic reactor components, such as ceramic fuels, moderators and cooling and cladding materials, are considered. Topics discussed are heterogeneous non-metallic fuels, metallic systems, aqueous systems and aqueous slurries of homogeneous fuels; coolants and moderators; light and heavy water reactions; liquid metal coolants; gaseous coolants; graphite moderator; and reactions of the canning or cladding materials such as aluminum and zirconium.—NSA. 15882

8.8 Group 8

8.8.3, 3.6.2, 3.6.6

Corrosion and Materials of Construction in the Plating Room. T. J. V. Cudbird. Proceedings, American Electroplaters' Soc., **44,** 91-94, discussion, 164 (1957).

Simple chemical corrosion, galvanic corrosion and concentration cell corrosion are three main methods of corrosion in plating room; methods to minimize them. Applications of steel, aluminum, lead, silicon-iron alloy, rubber, vinyls and plastics for tanks, tank linings, exhaust ducts and other uses in the plating room.—MR. 15780

8.8.5, 3.5.9

Determination of Corrosion Resistant Properties of Castings at Various Temperatures. (In French.) P. Detrez. Corrosion et Anticorrosion, **5,** 374-382 (1957) Dec.

Users should be very specific about working conditions and temperatures in ordering refractory castings and foundrymen should be explicit about the qualities of their castings. Causes for poor performance or cracking are graphitization, internal stresses (thermal), corrosion, fusion of phosphorus eutectic. Corrosion causes are variety of structural constituents, diversity of gas composition, different penetration speeds of gases into casting masses.—MR. 15759

8.8.5, 3.7.2, 6.3.19

The Influence of Aluminium Content on the Properties of Zinc Alloys for Die-Casting. L. A. J. Lodder and K. P. Scott. Fonderie, **139,** 367-377 (1957).

A brief account of zinc die-castings since 1910 with alloy compositions and the development of the Zamak series. The introduction of aluminum to the alloys and the zinc-aluminum system are outlined and the effects of aluminum in inhibiting attack on iron, on castability (in the range 0.6%), and on mechanical properties, hardness, and impact resistance are described. Ageing effects and intercrystalline corrosion, testing with steam, atmospheric exposure and effects of heat-treatment are tabulated and discussed.—MA. 15837

8.8.5, 6.2.5

Problems in Making Stainless Steel Castings. E. A. Schoefer. Paper before Niagara Frontier Regional Foundry Conf., Buffalo, October 24-25, 1957. Foundry, **86,** No. 4, 114-119 (1958) April.

Discusses problems besetting foundrymen producing high-alloy castings of iron-chromium and iron-chromium-nickel referred to as stainless steel and used for heat, corrosion and abrasion resistant applications. Problems of selecting right alloy from total of 107 different, closely controlled compositions reported by the industry, in order to provide highly specific combination of properties, is considered. Manufacturing problems of melting, mold and core making, cleaning costs, heat treatment, inspection and control are discussed. Table lists compositions of A.C.I. C and H series alloys. Ternary diagram shows location of most widely used grades.—INCO. 16005

8.8.5, 6.2.5

Manufacture and Properties of Stainless Sintered Steel. F. Eisenkolb. Stahl und Eisen, **78,** No. 3, 141-148 (1958) February 6.

Manufacture of 18-8 powders using following processes is described: intergranular disintegration, atomization, and jet atomization. Grain size grading, grain shape and pressing and sintering characteristics are considered. Effects of compacting pressure, sintering temperature, application of double pressing process, sintering time, and furnace gases as well as effect of addition of 0.1-0.3 boron, 1.0-1.5 red phorphorus and 0.1-0.6 phorphorus from ferro-phosphorus on specific weight, tensile strength and breaking elongation of austenitic sintered steels were investigated. Corrosion in n/2-hydrochloric acid and erosion by water of 18-8 as dependent on porosity of steels, and on duration of attack, are reported. Tensile strength, specific weight, and shrinkage of sintered compacts made of mechanically produced 18 chromium steel powder of varying grain sizes, were determined. Manufacture and mechanical properties of sintered compacts containing 25 nickel were reported. Tables, photomicrographs.—INCO. 15447

8.8.5, 8.4.5

Metallurgical Problems in Fabrication of Zirconium-Clad Fuel Elements for Pressurized Water Reactors. R. B. Gordon. Westinghouse Elec. Corp. Nuclear Science and Eng., **3,** No. 3, 232-249 (1958) March.

Discusses typical problems involving zirconium cladding alloys, fuel materials, and fuel element fabrication process. Development of Zircaloy-2 and Zircaloy-3 is considered. Problems relative to melting of sound homogeneous ingots free from objectional impurities are discussed. Although some zirconium-base alloys can be successfully produced by arc-melting, some of uranium-base alloys require induction melting. Crucible contamination problems may occur in higher melting alloys containing molybdenum and niobium. Early difficulties in compacting and sintering of uranium dioxide fuel have been eliminated. Zirconium-clad fuel elements of bonded type are produced by pack rolling or coextrusion of fuel and cladding. Dimensional problems relating to configuration of fuel core result from differences in plasticity of fuel and cladding; alloy additions to uranium fuel greatly improve core configuration of Zircaloy-clad elements. Unbonded fuel elements require close dimensional control of fuel and cladding components. Excellent welding characteristics of Zircaloy enable production of high-quality and closure welds. Much work remains to be done to achieve ultimate in high-temperature water corrosion resistance. Photomicrographs, cross sections of elements.—INCO. 15525

18 SIZES

to check electrolytic corrosion throughout your piping systems

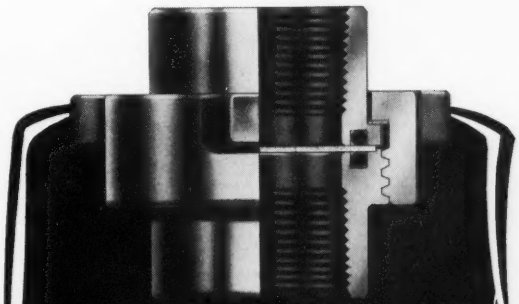

FIGURE 110
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Louis Ray—Farmington, N. M.
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L. P. Kinnear—Great Bend, Kansas
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8.8.2, 4.6.11

New Paths in Sea-Water Distillation. A. E. Williams. Chem. and Process Eng., **39**, No. 2, 55-58 (1958) Feb.

Description of an evaporating and distilling plant to serve Clifton Pier power station of Bahamas Electricity Corp. Equipment is made largely of Colclad stainless steel. Chemical treatment of phosphate type is used to retard scale formation on heating elements. Scale formation and corrosion in the plant are appreciably lowered by use of a closed feed system for boiler plant and use of de-aeration equipment. Different types of distillators, and development of useful by-products of sea water are described. Photos.—INCO. 15493

8.8.5

Which Finish for Zinc Die Castings. R. Stricklen. Allied Research Products, Inc. Product Eng., **29**, No. 9, 59-61 (1958) March 3.

A guide to mechanical, chemical, plating and painting methods is given. Electroplates applied to other metal products also work with zinc die castings but some change must be made in bath formula and method of application. Copper-nickel-chromium combination is by far the most common. Nickel plating solutions for coating zinc directly are available but generally give brittle deposits which are not bright. Chromium plate prevents tarnishing of nickel and adds wear resistance. Instead of chromium, deposits of brass, silver, or gold may be plated for decorative effects. These are nearly always followed by lacquer for wear resistance. Tables.—INCO. 15503

8.9 Group 9

8.9.1, 3.5.9

Materials for "Hot" Rocket Parts Must Withstand 1700 F Plus. R. C. Kopituk. Reaction Motors, Inc. Aviation Age, **28**, No. 7, 104-109 (1958) Jan.

Materials for liquid propellant rocket operation above 1700 F must be selected for their resistance to oxidation, corrosion and erosion, their strength, producibility and availability and finally their cost. Table lists various metals and ceramics used for gas generator (up to 1900 F), injector head (—420 to +2500 F), extension cone (1600-2500 F), uncooled combustion chamber (—65 to +4000 F in local hot areas), and regeneratively cooled combustion chamber (—65 to +2400 F in local hot areas). Materials include various high temperature alloys, Inconels, nickel, stainless steels and nickel base brazing alloys, among others. Injector partitions must be kept leakproof; tiniest leak would cause explosion of rocket. To make sure beads are safely seal-welded, they can be coated with acrylic slurry of high temperature nickel brazing alloy such as Coast 52 or 53 and fused at temperature above that for pinhole penetration by normal flow. Diagram shows hot parts of liquid propellant rocket.—INCO. 15749

8.9.2, 5.11

Corrosion of Car Bodies. K. A. Murrell. Corrosion Prevention and Control, **5**, No. 3, 49-54 (1958) March.

Review of corrosion of automobile bodies and consideration of preventive design and treatment. Experience has shown that worst design features in promotion of corrosion are sharp edges, crevices and sharp radiused concavities; difficulty is encountered in providing adequate protection. Improvements in design to eliminate water traps has reduced corrosion considerably. Investigations have shown that corrosion formed in sheltered areas is different from that formed on external surfaces exposed to weather. Use of phosphate pre-treatment and of metallic undercoatings (zinc, tin and zinc-aluminum alloy) is discussed. Vauxhall process employs special heavy antirust primer paint for undercoating which is complementary to normal finishing paint. Advances made in use of improved steels, aluminum and stainless steel for car bodies are analysed. Problems encountered in nickel plating of automobile components are considered. Bright nickel compared with dull nickel is far less effective as protection against corrosion. Nickel shortage gave greater incentive to development of bright anodized aluminum for trim. Disadvantage is softness of base metal which is more easily damaged by knocks and abrasive polishes than nickel-chromium finish.—INCO. 15675

8.9.2, 5.3.2

Finishing Industry Interviews. Four Attacks on the "Rusty Bumper" Problem. W. A. Wesley. Inco. Products Finishing, **22**, No. 10, 32-34 (1958) July.

Suggests following as possible practical solutions to problem of bumper corrosion: deposition of coatings with least objectional appearing breakdown; use of thicker chromium coatings; use of multiple nickel layers; and use of multiple nickel layers with one layer of metal quite different from nickel. Best combination for latter is nickel-strontium-nickel-chromium. Best way for plater to improve corrosion resistance of products right now is to double nickel thickness and multiply chromium thickness by four.—INCO. 16101

8.9.5

Newest Offshore Cruiser. J. Kingdon. Popular Boating, **3**, No. 5, 22-25, 76, 78, 80, 82 (1958) May.

Description of Frank Mayer's 49' offshore cruiser Natalie IV. Two stainless steel jalousies are located between doors of pilot-house for communication and ventilation. Beneath the lounge is a compartment that contains two 250 gallon Monel fuel tanks and two 100 gallon Monel water tanks. Monel tail shafts lead from V-drives through copper shaft tubes, bronze studding boxes and bronze struts with rubber bearings to 24 x 24 in. bronze three-bladed propellers. Engines are cooled with fresh water as the salt in sea water may induce corrosion and electrolysis, may cause leaks and cracks and may plug up the cooling passages. Four Elastomer resilient neoprene silencers muffled roar of engine to a minimum of mumble. These silencers are a brand-new product. They are extremely light in weight and since they are made of neoprene and thus can't rust or corrode, are guaranteed for 10 years. Double sink in galley is made of Monel. Photos.—INCO. 16075

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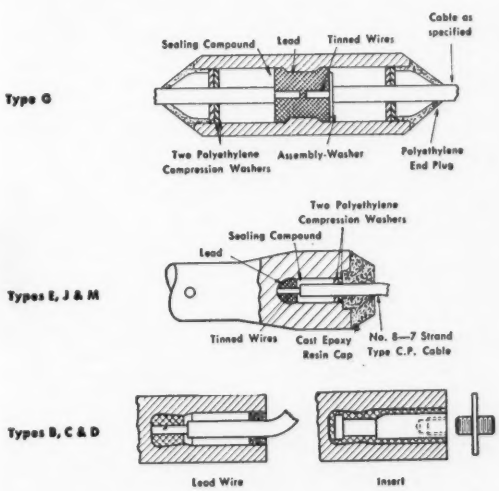

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D	2" x 60"	2.6	46	Ground Bed
E	3" x 60"	4.0	115	Salt Water
G	2" x 9"	0.4	5	Ducts
J	3" x 36"	2.5	80	Salt Water
M	2" x 60"	2.8	57	Salt Water

*Applicable also in fresh water service. Should not be used in ground beds without backfill.

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